FINAL SUPPORT SAMPLING PLAN

PART I - QUALITY ASSURANCE PROJECT PLAN

PART II- FIELD SAMPLING PLAN

PART III - HEALTH AND SAFETY PLAN

for the

ENGINEERING EVALUATION AND COST ANALYSIS
OF THE FORMER CELOTEX SITE
2800 South Sacramento Avenue
Chicago, IL 60623

Prepared for:

ALLIEDSIGNAL, INC.
MORRISTOWN, NEW JERSEY
and
THE CELOTEX CORPORATION
TAMPA, FLORIDA

MARCH 1997

Prepared by:

PARSONS ENGINEERING SCIENCE, INC.
1000 JORIE BOULEVARD, SUITE 250
OAKBROOK, IL 60521

Parsons ES Project No. 730577

FINAL SUPPORT SAMPLING PLAN

PART I

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for the

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Prepared for:
ALLIEDSIGNAL, INC., MORRISTOWN, NEW JERSEY
THE CELOTEX CORPORATION, TAMPA, FLORIDA

Prepared by:
PARSONS ENGINEERING SCIENCE, INC.
1000 JORIE BOULEVARD, SUITE 250
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Revision 1

MARCH 1997

Mona D. Sutherland, Parsons ES Project Manager	5/13/97 Date
Gregory Gibbons, P.E., Parsons ES QA/QC Manager	5/12/97 Date
Opal Davis-Johnson, Quanterra Environmental Services Project QA Officer	5/8/97 Date
David A. Paley, P.E., AlliedSignal Inc. Project Manager	4/16/97 Date
Legil Colburn, The Celotex Corporation Project Manager	4/28/97 Date
Thomas G. Williams, P.E., USEPA Region V Remedial Project Manager	5/19/97 Date
Richard Byvik, USEPA Region V Quality Assurance Reviewer	Date

PARSONS ENGINEERING SCIENCE, INC.
RESPONSE TO COMMENTS

DRAFT SUPPORT SAMPLING PLAN
FOR THE
ENGINEERING EVALUATION AND COST ANALYSIS (EE/CA)
OF THE
FORMER CELOTEX SITE
2800 South Sacramento Avenue
Chicago, Illinois

PART I QUALITY ASSURANCE PROJECT PLAN (QAPP)

I. PROJECT BACKGROUND

A. Section 2.7 TARGET COMPOUNDS pg 15/23, 2nd paragraph. If reactive cyanide and reactive sulfide exceed the EPA guidance levels, discuss some course of action.

The information generated from the disposal analyses will be evaluated during the remedial alternatives evaluation and screening stage of the EE/CA process to assess appropriate soil management and/or disposal requirements/limitations. Exceedance of the reactive cyanide and/or reactive sulfide guidance levels may result in the soils requiring some form of treatment such as alkaline chlorination (for cyanide) and/or oxidation of sulfides, should off-site management/disposal of Site soils be deemed an appropriate remedial measure. The classification of the soils may also be affected.

B. Section 2.7 TARGET COMPOUNDS pg 15/23, 3rd paragraph. The EE/CA Scope of Work (SOW), part B, Item 2, mentions that Sediment samples will be tested for Physical Properties. Please indicate these Physical Properties (particle size, particle size distribution, organic carbon, color, odor, etc.) and how they will be examined (laboratory or visual). Include the Physical Properties in Table 2.3 and Table 4.1.

The physical properties (odor, color, and lithology) of the sediment samples will be evaluated based on field observation (visual and olfactory). This information has been added to the discussion in the QAPP (Subsection 2.7, Table 2.3, Table 4.1, Subsection 8.2).

II. PROJECT ORGANIZATION AND RESPONSIBILITY

Section 3.2 Identify the Parsons ES Official(s) that will conduct the risk assessment.

Mr. Steven Noren will be responsible for conducting the main site risk assessment. Mr. Noren will coordinate with the Parsons ES project manager Ms. Mona Sutherland on all activities and issues related to the risk assessment. This information has been added to the QAPP as Subsection 3.2.2.5.

III. QUALITY ASSURANCE OBJECTIVES FOR MEASUREMENT DATA

A. Section 4.2.2, Section 4.7, and Table 4.1

1) For the Soil and Sediment matrices Field Rinseate Blank Samples are allowed, but not required.

No field rinseate blank samples will be collected or analyzed for soil and sediment samples. This amendment has been made throughout the QAPP (Table 4.1, and subsections 4.2.2, 4.7, and 9.2.3).

2) For the Soil and Sediment matrices Trip Blanks are allowed, but not required.

No trip blank samples will accompany or will be analyzed for soil and sediment samples. This amendment has been made throughout the QAPP (Table 4.1, and subsections 4.2.2, 4.7, and 9.2.3).

B. TABLE 4.3

1) Express the units in ug/kg, or mg/kg, rather than ppb.

The appropriate units have been added to Table 4.3.

2) For the PARAMETERS Flashpoint and pH the SOPs indicate that duplicate samples are analyzed. Stipulate some project RPD acceptance criteria.

The RPD acceptance criteria for both flash point and pH is 0 - 20 RPD. This change has been made to Table 4.3.

IV. CALIBRATION PROCEDURES AND FREQUENCY

A. Section 7.1 Specify the frequency for recalibration of pH and conductance meter, such as, every 4 hours and/or after every 10 samples.

The pH and specific conductance meter will be calibrated after approximately 4 hours of continuous operation or after every 10 groundwater samples are collected, whichever occurs first. Section 7.1 has been amended accordingly.

B. Section 7.1 pg 3/9 editorial. After the paragraph on thermometer calibration, insert the following: MicroTIP® Calibration

This change has been made as requested.

V. ANALYTICAL PROCEDURES

Section 8.2.1.3 and TABLE 8.1. Discuss how the physical properties of Sediments will be determined, and include them in TABLE 8.1.

Refer to the response to Comment I B.

VI. DATA REDUCTION, VALIDATION, AND REPORTING

Section 10.4 Parsons ES Data Validation and Reporting. The data package submitted by Quanterra should be sufficient to conduct the risk assessment.

This statement has been added to Subsection 10.4 as requested.

VII. APPENDIX B QUANTERRA LABORATORY STANDARD OPERATING PROCEDURES

A. TOXICITY CHARACTERISTIC LEACHING PROCEDURE (TCLP) SOP No. NC-IP-0005, Section 11.3.4.1.1. The reference to Section 11.3.7, perhaps should be 11.3.5.

The SOP has been changed accordingly. The updated SOP and SOP change form has been attached for your review.

B. INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS, METHOD 1010A AND METHOD 200.7 SOP No. CORP-MT-0001NC, Section 11.17.0. The reference to Appendix E, maybe, should be D.

An SOP change form has been created to note the error in the appendix reference. The SOP change form is attached for your review and should be incorporated with the CORP-MT-0001NC, Revision 1.1, SOP.

C. TOTAL ORGANIC CARBON (TOC) ANALYSIS FOR NON-WATERS SOP No. NC-WC-0018

1) This SOP is based on, "Methods of Soil Analysis, Walkley-Black." This method may be determining all of the Organic Carbon., i.e., Total Carbon, not just the TOC? Is this method comparable to other standard methods?

This method is comparable to the other methods and studies such as Schollenberger and Tinsley. The TOC analysis for soil samples is no longer present in the Standard Methods. The Walkley-Black method should be considered to give approximate or semi-quantitative estimates of organic carbon in soil because of the lack of an appropriate correction factor for each soil analyzed.

2) Section 1,1 and Section 17.11. The MDL specified in QAPP Table 4.3 for TOC is 13.11 ppb. The working linear range given here is 100 to 15000 mg/kg and the Reporting Limit is 100 mg/kg. Can this method achieve the project required MDL?

The MDL value of 13.11 ppb should be 13.11 ppm or mg/L. The ppb reference was a typographical error.

This value is statistically derived. Any values reported below the project-specific standard reporting limit of 50 mg/kg and above the MDL should be flagged as quantitation being "suspect." The increase in the possibility of reporting false positives or negatives are present when reporting between the RL and MDL. QA criteria such as calibration curves and method blanks are based on the standard reporting limit unless requested otherwise.

3) ***Section 11.3.2. More than one aliquot, 4 are recommended, of each soil sample should be tested and the average reported.***

The averaging of four aliquots is not part of the Walkley-Black procedure. The method states to transfer a weighed soil amount not in excess of 10 grams into an Erlenmeyer flask.

4) ***This procedure does not include heating as indicated in Section 4.3, Inorganic Carbon will be included in the sample results.***

Heating to remove interferences should have been removed from the SOP. The laboratory switched from Standard Methods. Sixth Edition version of the method to Soil of Method Analysis. Section 4.3 was left in inadvertently from the previous version of the SOP. The SOP will be revised accordingly. The Walkley-Black method in Soil of Method Analysis does not require "heating" of the sample(s).

5) ***Section 12.1. According to the formula sample results are not being reported on a dry-weight basis. The results will be Total Carbon.***

All soil samples are reported on a dry-weight basis. Percent moisture is determined for all soil samples upon receipt and the result is entered independent of the requested sample analysis. The TOC analyst reports the Total Organic result in the LIMs, and the dry-weight result is calculated and reported to the client.

VIII. APPENDIX C CLP, TCL, AND TAL CONTRACT REQUIRED QUANTITATION LIMITS

There are 2 sets of tables, delete 1. Pages C-4 and C-6 are missing. Please include.

All changes have been made as requested, and a complete set of the TCL and TAL quantitation limits is enclosed herein.

IX. PART II FIELD SAMPLING PLAN

A. Section 5.5 and Section 6.4

1) ***Specify the upgradient and downgradient inlet locations. Is the upgradient location within the inlet? Is the downgradient location in the Chicago Sanitary and Ship Canal? A map of the locations would be helpful.***

The upgradient and down gradient sediment samples will be collected from the Chicago Sanitary and Ship Canal. Figure 5.2 shows the approximate locations where these sediment samples will be collected.

2) ***Describe how sediment samples for Physical Properties are collected and characterized. Include these in Table 5.1 and Table 6.1.***

Refer to the response to Comment I B.

B) Section 6.2 pg 3/13, 2nd paragraph, typo. The reference to Subsection 6.6, should be 6.7.

This correction has been made.

C) Section 6.4 pg 7/13, 1st paragraph, type. The reference to Subsection 6.5, should be 6.6.

This correction has been made.

D) Section 6.5.3

1) Please emphasize that VOC samples are not to be filtered.

A statement indicating that VOC samples will not be filtered has been added to Subsection 6.5.3.

2) See Comment III A above.

No trip blank samples will accompany or will be analyzed for soil and sediment samples. This amendment has been made throughout the FSP (Subsection 6.5, Table 6.1).

E) Section 7.2. Provide copies of the Sample Labels and Custody Seals.

An example of the sample container label and the custody seal that will be used during this phase of field investigation of the main site has been provided as Figure 7.1.

X. PART III HEALTH AND SAFETY PLAN

Section 9. Unless already known, it would be helpful to know in advance if Mt. Sinai Hospital will accept contaminated patients.

Mt. Sinai Hospital will accept patients who are wearing contaminated clothing or are otherwise contaminated. The field team leader is required to call the hospital's emergency room immediately if it is known that this situation has occurred. The phone call will allow the hospital time to prepare for the arrival of the potentially "contaminated" patient. Section 9.1 has been amended to reflect this information.

FINAL SUPPORT SAMPLING PLAN
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Revision 1

MARCH 1997

Mona D. Sutherland, Parsons ES Project Manager	Date
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TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

TABLE OF CONTENTS

TABLE OF CONTENTS

Based on information currently available on the Site, Parsons ES will target PAHs as the primary contaminants of concern. PAH data will be generated for all soil samples. Furthermore, as part of the overall Site evaluation process, all soil samples will also be analyzed for eight metals (arsenic, barium, cadmium, chromium, lead, mercury, selenium, and silver) and pH.

To determine whether petroleum-based contamination is present across the Site, or whether it is limited to localized areas in the vicinity of the former tank storage areas, all soil samples will be analyzed for volatile organic compounds (VOCs). In addition, all surface soil samples and approximately 10 percent of the subsurface soil samples collected during this investigation will be analyzed for semivolatile organic compounds (SVOCs) and pesticides/polychlorinated biphenyls (PCBs), to confirm that these constituents do not pose a problem sitewide.

Approximately ten percent of the soil samples will be analyzed for total organic carbon (TOC) and disposal parameters (the eight metals via the toxicity characteristic leaching procedure [TCLP], TCLP VOCs, reactive cyanide, reactive sulfide, flash point, and sulfur). Geotechnical analyses (porosity, permeability, grain size, bulk density, and BTU content) will also be run on approximately ten percent of the soil samples. The TOC, disposal parameter and geotechnical data will be used during the remedial alternatives evaluation stage to provide important preliminary insight into the potential issues associated with both *in situ* and off-site management of Site waste.

All groundwater samples will be analyzed for VOCs, SVOCs, pesticides/PCBs, the eight metals (on filtered and unfiltered samples), and cyanide, and will be field screened for specific conductance, temperature, and pH. All sediment samples will be analyzed for PAHs, the eight metals, cyanide, and pH. The physical characteristics of the sediments (odor, color, and lithology) will be assessed in the field by the Parsons ES field geologist via visual and olfactory observations. Table 2.3 presents a summary of the investigative

TABLE 2.3
INVESTIGATIVE SAMPLE SUMMARY

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

		Analytical Parameters and Number of Investigative Samples									
		VOCs	PAHs	SVOCs	8 Metals	Cyanide	Pesticides/ PCBs	Disposal Parameters	Geotech Parameters	pH	Total Organic Carbon
Site Sector	**Sample Depth**										
Sec A (Soil)	0 - 6"	14	0	14	14	14	14	0	2	14	0
(14 Locations)	1 -2'	14	12	2	14	14	2	2	2	14	2
	Deep	14	12	2	14	14	2	2	2	14	2
Sec B (Soil)	0 - 6"	6	0	6	6	6	6	0	1	6	0
(6 Locations)	1 - 2'	6	5	1	6	6	1	1	1	6	1
	Deep	6	5	1	6	6	1	1	1	6	1
Sec C (Soil)	0 - 6"	3	0	3	3	3	3	0	0	3	0
(3 Locations)	1 - 2'	3	2	1	3	3	1	1	0	3	1
	Deep	3	2	1	3	3	1	1	0	3	1
Sec D (Soil)	0 - 6"	2	0	2	2	2	2	0	0	2	0
(2 Locations)	1 - 2'	2	0	2	2	2	1	1	0	2	1
	Deep	2	0	2	2	2	1	1	0	2	1
Sec E (Soil)	0 - 6"	4	0	4	4	2	4	0	1	4	0
(4 Locations)	1 - 2'	4	3	1	4	4	1	1	1	4	1
	Deep	4	3	1	4	4	1	1	1	4	1
Sec F (Soil)	0 - 6"	6	0	6	6	6	6	0	1	6	0
(6 Locations)	1 - 2'	6	5	1	6	6	1	1	1	6	1
	Deep	6	5	1	6	6	1	1	1	6	1
Sec G (Soil)	0 - 6"	5	0	5	5	5	5	0	1	5	0
(5 Locations)	1 - 2'	5	4	1	5	5	1	1	1	5	1
	Deep	5	4	1	5	5	1	1	1	5	1

TABLE 2.3
INVESTIGATIVE SAMPLE SUMMARY

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

		Analytical Parameters and Number of Investigative Samples									
		VOCs	PAHs	SVOCs	8 Metals	Cyanide	Pesticides/ PCBs	Disposal Parameters	Geotech Parameters	pH	Total Organic Carbon
Site Sector	**Sample Depth**										
Background (Soil)	0 - 6"	1	0	1	1	1	1	0	1*	1	1
	1 - 2'	1	0	1	1	1	1	0	1*	1	1
	Deep	1	0	1	1	1	1	0	1*	1	1
Unassigned (soil)	0-6	5	10	0	10	10	5	[illegible]	0	10	5
(10 locations)	1-2	5	10	0	10	10	5	5	0	10	5
	Deep	5	10	0	10	10	5	5	0	10	5
SOILS SUBTOTAL		138	92	61	153	153	74	31	18	153	34
Sediment (in inlet)	Not Applicable	0	1	0	1	1	0	0	0	1	0
Sediment (up-gradient)	Not Applicable	0	1	0	1	1	0	0	0	1	0
Sediment (down-gradient)	Not Applicable	0	1	0	1	1	0	0	0	1	0
SEDIMENT SUBTOTAL		0	3	0	3	3	0	0	0	3	0
Groundwater (4 Site Wells)	Not Applicable	4	0	4	4	4	4	0	0	4	0

Notes

- Site sectors are shown on Figure 5.1 of the Field Sampling Plan (Part 2 of the SSP).
- Metals analysis will be performed on both filtered and unfiltered groundwater samples collected from each well point.
- The deep soil sample will be collected at a depth of 4 to 6 feet below ground surface unless sample material collected at a deeper sample interval exhibits indications of greater soil contamination below the 4- to 6-foot interval.
- Quality control samples are described and listed in Subsection 6.5 of the Field Sampling Plan (Part 2 of the SSP).
- Disposal parameters refer to the following analyses: TCLP VOCs, TCLP metals, reactive cyanide, reactive sulfide, flashpoint, and sulfur.
- Geotechnical parameters refer to the following analyses: porosity, permeability, bulk density, grain size, and BTU content.
- *Refers to grain size analysis only. The soil subtotal does not include these samples.

samples that will be collected during this field investigation and the associated analytical parameters.

The sampling program outlined in this SSP has been designed (1) to minimize the need for a second phase of field sampling prior to the preparation of the EE/CA report, and (2) to facilitate the collection of anticipated necessary information needed to complete the EE/CA for the Site. Per the AOC, a Data Report will be prepared based on the analytical data generated from this field sampling program. The Data Report will be submitted for USEPA Region V review. If the data generated from this first phase of field investigation is deemed sufficient for site characterization, the Respondents and Parsons ES will proceed with the preparation of the main site risk assessment.

2.8 INTENDED DATA USAGE

The data generated from this investigation will be used to preliminarily characterize contaminant sources and pathways, to determine the potential extent of contamination, and to assess routes of current off-site contaminant migration, if any. This information will then be used (1) to delineate the approach for future Site remedial activities, and (2) in the development of the main Site risk assessment and EE/CA reports.

Field screening data generated from the headspace evaluation of soil samples and the data generated from other contaminant indicators (visual and olfactory observations) will be used as a qualitative guide to determining the relative potential degree of contaminant levels in the soil samples, thereby enabling the most impacted soil samples to be chosen for analysis.

The shallow subsurface soils analytical data will provide information on source characterization. The vertical extent of contamination will be assessed based on the analytical data generated from the analysis of the deep subsurface soil samples. The horizontal extent of contamination will be determined based on the analytical data generated from the spatially distributed soil boring locations. The determination of Site property

boundaries through a deed search/surveying, and the placement of soil boring locations close to these boundaries will provide information on whether Site contamination extends to these boundaries. In addition, the analytical data from the groundwater screening program will provide some insight into the groundwater conditions below the Site.

The data generated from TOC and pH analyses of soil samples, the hydrogeologic information resulting from the groundwater screening program (groundwater flow direction, specific conductance, pH), and information from the topographic survey of the Site will facilitate the characterization of Site contaminant pathways. Contaminant pathways will also be assessed through the sampling of any on-site erosional gullies, ditches, etc., that are present on site during the period the sampling program is executed.

The data from soil disposal parameter and geotechnical analyses will be used during the remedial alternatives evaluation process to preliminarily assess the hazardous waste characteristics of the sampled materials and to facilitate a preliminary evaluation of the waste management requirements and remedial options for the material.

The main site risk assessment will be developed and the remedial action alternatives will be assessed based on the analytical data and the qualitative information generated from this EE/CA sampling program. As specified in the Site EE/CA Work Plan (January 1997), the main site risk assessment will evaluate on-site contamination and the associated risks to human health and the environment within the context of future commercial or industrial development of the Site.

2.9 DATA QUALITY OBJECTIVES AND CRITERIA FOR MEASUREMENT DATA

The Data Quality Objective (DQO) process is a series of planning steps based on a scientific method that is designed to ensure that the type, quality, and quantity of environmental data used in decision making are appropriate for the intended application. The steps of the DQO process are included in Figure 1-1 of the USEPA Region V Model QAPP (May 1996).

The DQOs are qualitative and quantitative statements derived from the outputs of each step of the DQO process that:

- Clarifies the study objectives.
- Defines the most appropriate type of data to collect.
- Determines the most appropriate conditions from which to collect the data.

The DQOs are then used to develop a scientific and resource-effective sampling program design. The DQO process allows decision makers to define their data requirements and acceptable levels of decision during the planning stage, before any data are collected. DQOs should be based on the seven step process described in the USEPA QA/G-4 (September 1994) document and outlined in Figure 1-1 of the USEPA Region V Model QAPP (May 1996).

2.10 SAMPLE NETWORK DESIGN AND RATIONALE

The sample network design and rationale is described in Section 5 of the FSP (Part 2 of the SSP).

2.11 PROJECT SCHEDULE

2.11.1 Anticipated Date of Project Mobilization

The project schedule for the overall EE/CA, including activities associated with the SSP and the field sampling program, is summarized in Table 2.4. The summary table contains four task information columns alongside the task name description that specify (1) task duration in days ["d" represents business days and "ed" represents calendar days], (2) the anticipated start date for a task, (3) the anticipated finish date for each task, and (4) task predecessor information which identifies the constraints and limitations in schedule flexibility. Based on the assumptions used in the development of this schedule, the earliest date on which the field sampling program can commence is 2 May 1997.

2.11.2 TASK BAR CHART AND ASSOCIATED TIME FRAMES

The project schedule bar chart, prepared in Gantt chart format, is presented as Figure 2.5

TABLE 2.4
SUMMARY OF PROJECT SCHEDULE

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE
CHICAGO, ILLINOIS

ID	Task Name	Duration	Start	Finish	Predecessors
1	Effective Date of Consent Order	0	11/1/96	11/1/96	
2	**Draft Work Plan (WP)**	**17**	**11/1/96**	**11/26/96**	**1**
3	Prep of Draft Work Plan	12	11/1/96	11/13/96	
4	Internal Review of Draft WP	13	11/13/96	11/26/96	3
5	Submittal of Draft WP to EPA	0	11/26/96	11/26/96	4
6	**EPA Review of Draft WP**	**31**	**11/26/96**	**1/7/97**	
7	Review Period	31	11/26/96	1/7/97	5
8	**Final WP**	**10**	**1/8/97**	**1/21/97**	
9	Prep of Final WP	6	1/8/97	1/15/97	7
10	Internal Review of Final WP	4	1/16/97	1/21/97	9
11	Submittal of Final WP to EPA	0	1/21/97	1/21/97	10
12	**EPA Review of Final WP**	**30**	**1/22/97**	**3/4/97**	**11**
13	Review/Approval Period	30	1/22/97	3/4/97	
14	**Residential Conceptual WP**	**60**	**3/5/97**	**5/27/97**	
15	Prep of Draft WP	45	3/5/97	5/6/97	13
16	Internal Review of Draft WP	15	5/7/97	5/27/97	15
17	Submittal of Draft WP to EPA	0	5/27/97	5/27/97	16
18	**EPA Review of Draft Residential WP**	**14**	**5/28/97**	**6/16/97**	
19	Review/Approval Period	14	5/28/97	6/16/97	17
20	**Response to Comments on Res. Draft WP**	**88**	**6/17/97**	**10/16/97**	
21	Prep and Submittal of Response	14	6/17/97	7/4/97	19
22	EPA Review of Response	14	7/7/97	7/24/97	21
23	Agreement on Draft Res. WP Changes	60	7/25/97	10/16/97	22
24	**Finalize Residential Conceptual WP**	**75**	**10/17/97**	**1/29/98**	
25	Prep of Final Plan	30	10/17/97	11/27/97	23
26	Internal Review of Final Plan	30	11/28/97	1/8/98	25
27	Submittal of Final Plan to EPA	0	1/8/98	1/8/98	26
28	EPA Review of Final Plan	14	1/9/98	1/28/98	27
29	EPA Approval Period	1	1/29/98	1/29/98	28
30	**Draft SSP**	**35**	**11/26/96**	**1/14/97**	**2**
31	Prep of Draft SSP	35	11/26/96	12/31/96	
32	Internal Review of Draft SSP	14	12/31/96	1/14/97	31
33	Submittal of Draft SSP to EPA	0	1/14/97	1/14/97	32
34	**EPA Review of Draft SSP**	**26**	**1/14/97**	**2/18/97**	**30**
35	Review Period	26	1/14/97	2/18/97	
36	**Response to Comments on Draft SSP**	**21**	**2/19/97**	**3/19/97**	**34**
37	Prep and Submittal of Response	14	2/19/97	3/10/97	
38	EPA Review of Response	7	3/11/97	3/19/97	37
39	**Final SSP**	**14**	**3/20/97**	**4/8/97**	**36**
40	Prep of Final SSP	7	3/20/97	3/28/97	38
41	Internal Review of Final SSP	7	3/31/97	4/8/97	40
42	Submittal of Final SSP to EPA	0	4/8/97	4/8/97	41
43	**EPA Review of Final SSP**	**2**	**4/9/97**	**4/10/97**	
44	Review/Approval Period	2	4/9/97	4/10/97	42
45	**EE/CA Sampling Program**	**36**	**4/11/97**	**5/30/97**	
46	In-house prep activities	14	4/11/97	4/30/97	44
47	Mob to field	1	5/1/97	5/1/97	46
48	Execute Sampling Program	21	5/2/97	5/30/97	47
49	Demob from field	0	5/30/97	5/30/97	48
50	**Data Generation**	**62**	**5/6/97**	**7/30/97**	
51	Lab Analysis	55	5/6/97	6/30/97	48SS+2d,49FF+30ed
52	Data Validation	40	6/5/97	7/30/97	51SS+30ed
53	Data Report	51	7/11/97	9/19/97	
54	Data Reduction,Tabulation, Evaluation	21	7/11/97	8/8/97	51FS+2d,52FF+7d
55	Data Report Prep	35	7/15/97	9/1/97	54FF+10d,49SS+7d

TABLE 2.4
SUMMARY OF PROJECT SCHEDULE

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE
CHICAGO, ILLINOIS

ID	Task Name	Duration	Start	Finish	Predecessors
56	Data Report Internal Review	14	9/2/97	9/19/97	55
57	Submittal of Data Report to EPA	0	9/19/97	9/19/97	56
58	**EPA Review of Data Report**	**30**	**9/22/97**	**10/31/97**	
59	Review Period	30	9/22/97	10/31/97	57
60	**Response to Comments on Data Report**	**28**	**11/3/97**	**12/10/97**	
61	Prep and Submittal of Response	14	11/3/97	11/20/97	59
62	EPA Review of Response	14	11/21/97	12/10/97	61
63	**Finalize Data Report**	**14**	**12/11/97**	**12/30/97**	
64	Prep of Final Data Report	7	12/11/97	12/19/97	62
65	Internal Review of Final Data Report	7	12/22/97	12/30/97	64
66	Submittal of Final Data Report to EPA	0	12/30/97	12/30/97	65
67	**EPA Review of Final Data Report**	**14**	**12/31/97**	**1/19/98**	
68	Review/Approval Period	14	12/31/97	1/19/98	66
69	**Main Site Risk Assessment**	**75**	**9/29/97**	**1/9/98**	
70	Prep of Draft RA	45	9/29/97	11/28/97	57FS+5d
71	Internal Review of Draft RA	30	12/1/97	1/9/98	70
72	Submittal of Draft RA Report to EPA	0	1/9/98	1/9/98	71
73	**EPA Review of Draft RA**	**30**	**1/12/98**	**2/20/98**	
74	Review Period	30	1/12/98	2/20/98	72
75	**Response to Comments on Draft RA**	**42**	**2/23/98**	**4/21/98**	
76	Prep and Submittal of Response	14	2/23/98	3/12/98	74
77	EPA Review of Response	14	3/13/98	4/1/98	76
78	Agreement on Draft RA Changes	14	4/2/98	4/21/98	77
79	**Finalize RA**	**29**	**4/22/98**	**6/1/98**	
80	Prep of Final RA	10	4/22/98	5/5/98	78
81	Internal Review of Final RA	4	5/6/98	5/11/98	80
82	Submittal of Final RA to EPA	0	5/11/98	5/11/98	81
83	EPA Review of Final RA	14	5/12/98	5/29/98	82
84	EPA Approval of Final RA	1	6/1/98	6/1/98	83
85	**Remedial Objectives**	**14**	**6/2/98**	**6/19/98**	
86	Cleanup Objectives from EPA	14	6/2/98	6/19/98	84
87	**Draft EE/CA Report**	**59**	**5/11/98**	**7/30/98**	
88	Prep of Draft Report	45	5/11/98	7/10/98	86FS-30d
89	Internal Review of Draft EE/CA Report	14	7/13/98	7/30/98	88
90	Submittal of Draft EE/CA Report to EPA	0	7/30/98	7/30/98	89
91	**EPA Review of Draft EE/CA Report**	**30**	**7/31/98**	**9/10/98**	
92	Review Period	30	7/31/98	9/10/98	90
93	**Response to Comments on Draft EE/CA Report**	**28d**	**9/11/98**	**10/20/98**	
94	Prep and Submittal of Response	14d	9/11/98	9/30/98	92
95	EPA Review of Response	14d	10/1/98	10/20/98	94
96	**Final EE/CA Report**	**28d**	**10/21/98**	**11/27/98**	
97	Prep of Final EE/CA Report	10d	10/21/98	11/3/98	95
98	Internal Review of Final EE/CA Report	4d	11/4/98	11/9/98	97
99	Submittal of EE/CA Report to EPA	0d	11/9/98	11/9/98	98
100	EPA Review of Final EE/CA Report	14d	11/10/98	11/27/98	99
101	**Community Review of EE/CA Report**	**44d**	**12/14/98**	**2/11/99**	
102	Community Review and Comment Period	30d	12/14/98	1/22/99	100FS+10d
103	EPA Responsiveness Summary Prep	14d	1/25/99	2/11/99	102
104	**Approval of Final EE/CA Report**	**1d**	**3/4/99**	**3/4/99**	
105	EPA Approves Final EE/CA Report	1d	3/4/99	3/4/99	103FS+14d

noncompliance issues are identified, the QA/QC manager will communicate these findings to the project manager. The project manager will inform the QA/QC manager of the corrective measures that will be implemented to correct the problem. The QA/QC manager will document all noncompliance issues and the corrective measures taken, and may perform follow-up actions (if deemed necessary) to confirm that the corrective measures were effective in rectifying the issue. The QA/QC manager will not be involved with the day-to-day activities of the project to ensure an unbiased project review.

3.2.2.4 Data Management

Ms. Christine J. Shield will be responsible for project data management. In this role, Ms. Shields will track and oversee the review of all field and laboratory data. Ms. Shields will notify the Parsons ES project manager immediately of any QC problems associated with the analytical data and will coordinate with the laboratory to ensure the problems are rectified/addressed appropriately.

3.2.2.5 Risk Assessment

Mr. Steven Noren will be responsible for conducting the main site risk assessment. Mr. Noren will liaison directly with the Parsons ES project manager on all activities and issues related to this risk assessment.

3.2.2.6 USEPA Project Coordination

The USEPA Region V will be performing oversight of all activities performed for the Site EE/CA study. The USEPA remedial project manager is Mr. Thomas Williams, P.E. Mr. Williams will be the point of contact between the Respondents (and/or Parsons ES) and the USEPA Region V. All deliverables provided to the USEPA for review and approval will be sent to the attention of Mr. Williams.

project manager. Ms Horton will report directly to the Parsons ES project manager on a daily basis (or more frequently if deemed necessary) during the execution of the field sampling program.

3.4.2 Project Health and Safety Officer

Ms. Kim Horton will also be health and safety officer for the project. As health and safety officer, Ms. Horton's duties will include ensuring that all Site personnel are conversant with the health and safety requirements for the project and are executing them as outlined in the HASP, and will include being lead documentor of the project's safety and health issues. Because of the remote possibility for Level B conditions, Mr. Brian Powell, Certified Industrial Hygienist of the Parsons ES Syracuse office, will assist Ms. Horton if Level B protection is required. Mr. Powell or his designee will perform a site audit, should the work actually require Level B protection. The deputy health and safety officer will be Ms. Karen Carlisle. Ms. Carlisle will execute Ms. Horton's health and safety activities in her absence or if requested to do so by Ms. Horton.

3.4.3 Field Team Members

The anticipated field team will include Ms. Horton, field team leader; Ms. Karen Carlisle, geologist; Mr. Christopher Donohoe, junior engineer; and Mr. Randall Christy, technician. The field team will continue with the project through the preparation of the draft data report. During field activities, all field team members will have key QC responsibilities for ensuring all work is performed in accordance with the approved protocols outlined in the final SSP.

3.5 LABORATORY RESPONSIBILITIES

3.5.1 Overview

Quanterra Environmental Services, Inc. (Quanterra) located in North Canton, Ohio, will perform all chemical analyses for VOCs, SVOCs, PAHs, pesticides/PCBs, 8 total metals, cyanide, pH [soil and sediments], TOC, TCLP VOCs, TCLP metals, reactive

cyanide, reactive sulfide, flash point, sulfur, and the geotechnical analysis for BTU content. This subsection provides an overview of the Quanterra QA organization and identifies the project-specific QA organization, key personnel and responsibilities, and the lines of communication among these persons. The project-specific laboratory organization and key personnel roles and titles were presented previously on Figure 3.1. The remaining geotechnical parameter analyses (porosity, permeability, bulk density, and grain size) will be performed by Hanson Engineers, Inc. located in Springfield, Illinois. Unless otherwise specified, all generic references within this QAPP to "laboratory" pertain solely to Quanterra.

3.5.2 Quanterra Quality Assurance Organization

Quanterra has both quality and QA programs. Quality programs address all aspects of the product Quanterra delivers; therefore, they evaluate and improve, for example, vendor supplies, computer systems, laboratory processing operations, and personnel training. The QA program examines and defines test method QC and regulatory requirements, and directs and performs systems and performance audits.

There is overlap between the two programs, and at the corporate level, the QA efforts are administered by the chief operating officer (COO), who is also responsible for the quality programs. The COO reports directly to the president and CEO of Quanterra. Reporting to the COO is the corporate director of QA, who is responsible for QA. The corporate director of QA has overall responsibility for the analytical QA aspects of Quanterra. The corporate director of QA operates independent of all areas generating analytical data to ensure objectivity in the evaluation of laboratory analytical operations and QA issues.

Each laboratory facility has a quality assurance manager who monitors the day-to-day QA activities of that facility and reports directly to the director of QA and indirectly to the facility laboratory director. Finally, all scientists, analysts, and supervisory personnel within the laboratory and in support services play a vital role in ensuring that the general

TABLE 4.1
SUMMARY OF INVESTIGATIVE AND QUALITY CONTROL SAMPLES

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Matrix	Analyses	Estimated Total No. of Investigative Samples	Estimated No. of Field Duplicate Samples	Estimated No. of Field Rinseate Blank Samples	Estimated No. of MS/MSD Samples[1]	Matrix Total[2]
Soil						
	VOCs	138	14	0	7	152
	PAHs	92	10	0	5	102
	SVOCs	61	7	0	4	68
	8 Metals[3]	153	16	0	8	169
	Cyanide	153	16	0	8	169
	Pesticides/PCBs	74	8	0	4	82
	Disposal Parameters[4]	31	0	0	0	31
	Geotechnical Parameters[5]	21	0	0	0	21
	pH	153	0	0	0	153
	TOC	38	0	0	0	38
Sediment					0	
	VOCs	0	0	0	0	0
	PAHs	3	1	0	1	4
	SVOCs	0	0	0	0	0
	8 Metals[3]	3	1	0	1	4
	Cyanide	3	1	0	1	4
	Pesticides/PCBs	0	0	0	0	0
	Disposal Parameters[4]	0	0	0	0	0
	Physical[6] Parameter	3	1	0	0	4
	pH	3	0	0	0	3
	TOC	0	0	0	0	0

TABLE 4.1
SUMMARY OF INVESTIGATIVE AND QUALITY CONTROL SAMPLES

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Matrix / Analyses	Estimated Total No. of Investigative Samples	Estimated No. of Field Duplicate Samples	Estimated No. of Field Rinseate Blank Samples	Estimated No. of MS/MSD Samples[1]	Matrix Total[2]
Groundwater					
VOCs	4	1	1	1	6
PAHs	0	0	0	0	0
SVOCs	4	1	1	1	6
8 Metals[3] (filtered)	4	1	1	1	6
8 Metals[3] (unfiltered)	4	1	1	1	6
Cyanide	4	1	1	1	6
Pesticides/PCBs	4	1	1	1	6

Notes:

(1) Matrix spike/matrix spike duplicate (MS/MSD) samples are not additional samples, but investigative samples on which MS/MSD analyses are performed. This column also includes spike/duplicate or MS/MSD samples for inorganic analyses.

(2) The matrix total does not reflect MS/MSDs as additional samples. Trip blank samples are not included in the matrix total. One trip blank will be included with each shipment of VOC groundwater samples. Trip blanks will only be analyzed for VOCs.

(3) The 8 metals are arsenic, barium, cadmium, chromium, mercury, selenium, silver, and lead.

(4) Disposal parameters refer to TCLP 8 metals, TCLP VOCs, reactive cyanide, reactive sulfide, flash point, and sulfur.

(5) Geotechnical parameters refers to porosity, permeability, bulk density, grain size, and BTU content. * Three of these soil samples will be analyzed for grain size only.

(6) Physical parameters refers to odor, color, and lithology. These parameters will be determined in the field by the field geologist via olfactory and visual observations.

4.1.3 Laboratory Precision Objectives

Precision in the laboratory is assessed through the calculation of relative percent differences (RPDs) and relative standard deviations (RSDs) for three or more replicate samples. The equations to be used for precision in this project can be found in Section 13 of this QAPP. The precision requirements for organic analyses performed according to the current USEPA Contract Laboratory Program (CLP) Statement of Work OLM03.1, and for inorganic analyses performed according to USEPA CLP Statement of Work ILM04.0 are specified therein. The precision control limits for the remaining parameters are summarized in Table 4.3.

4.2 ACCURACY

4.2.1 Definition

Accuracy is the degree of agreement between an observed value and an accepted reference value.

4.2.2 Field Accuracy Objectives

Accuracy in the field is assessed through the use of field and trip blank samples (aqueous samples only) and through the adherence to all sample handling, preservation, and holding time requirements. Table 4.1 presents the number of field blank samples estimated for this field program and the frequency of collection of trip blanks. Sample handling, preservation, and holding times are discussed in the FSP. For field screening analyses associated with pH, specific conductance, and temperature measurements, accuracy requirements are summarized in Table 4.2.

4.2.3 Laboratory Accuracy Objectives

Laboratory accuracy is assessed through the analysis of matrix spike (MS) or standard reference materials (SRMs), and the determination of percent recoveries. The equation to be used for accuracy in this field program is discussed in Section 13 of this QAPP. The accuracy

TABLE 4.3
QC OBJECTIVES FOR NON-CLP PARAMETERS

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

PARAMETERS	MS/MSD	RPD	LCS	MDL(mg/L)	SPIKE CONCENTRATIONS
TCLP Metals	50-150	0-20	50-150	*See Below	**See Below
Reactive Cyanide	NA	0-20	NA	NA	NA
Reactive Sulfide	NA	0-20	NA	NA	NA
Flashpoint	NA	0-20	NA	NA	NA
pH	NA	0-20	NA	NA	NA
Total Organic Carbon	NA	NA	75-125	13.11	NA
Sulfur	±25%	0-20	80-120	(2)	5,000 mg/kg Sodium Sulfite(3)

ELEMENT	*MDL (ug/L)	**SPIKE CONCENTRATIONS (ug/L)
Arsenic	15.86	5,000
Barium	1.53	50,000
Cadmium	2.99	1,000
Chromium	4.75	5,000
Lead	32.40	5,000
Selenium	63.09	1,000
Silver	2.17	1,000
Mercury	0.0197	5

PARAMETERS	MS/MSD / RPD	LCS/LCSD / RPD	MDL (ug/L)	SPIKE CONCENTRATIONS	
TCLP Volatiles				MS/MSD (mg/L)	LCS (mg/L)
Benzene	37-151 / 21	73-118 / 15	0.26	0.25	0.10
Methyl ethyl ketone			8.0		
Carbon tetrachloride			0.37		
Chlorobenzene	37-160 / 19	76-120 / 15	0.39	0.25	0.10
Chloroform			0.45		
1,2-Dichloroethane			0.23		
1,1-Dichloroethylene	10-234 / 27	60-109 / 21	0.55	0.25	0.10
Tetrachloroethylene			0.36		
Trichloroethylene	71-157 / 20	75-119 / 13	0.48	0.25	0.10
Vinyl chloride			0.32		
Toluene (1)	47-150 / 15	76-119 / 15	NA	0.25	0.10

(1) Toluene is not reported on the TCLP list of analytes, but is on the standard spiking list.

(2) MDL will be established as part of the analytical procedure.

(3) This is the initial spiking concentration. Amendments may be made depending on final MDL.

requirements for organic analyses performed according to the current USEPA CLP Statement of Work OLM03.1, and for inorganic analyses performed according to USEPA CLP Statement of Work ILM04.0 are specified therein. The accuracy control limits for the remaining parameters are summarized in Table 4.3.

4.3 COMPLETENESS

4.3.1 Definition

Completeness is a measure of the amount of valid data obtained from the measurement system compared to the amount that was expected to be obtained under normal conditions.

4.3.2 Field Completeness Objectives

Field completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 13 of this QAPP. Field completeness for this project will be equal to or greater than 90 percent.

4.3.3 Laboratory Completeness Objectives

Laboratory completeness is a measure of the amount of valid measurements obtained from all the measurements taken in the project. The equation for completeness is presented in Section 13 of this QAPP. Laboratory completeness for this project will be equal to or greater than 90 percent.

4.4 REPRESENTATIVENESS

4.4.1 Definition

Samples must be representative of the environmental media being sampled. Representativeness expresses the degree to which data accurately and precisely represents a characteristic of a population, parameter variations at a sampling point, a process condition, or an environmental condition.

4.4.2 Measures to Ensure Representativeness of Field Data

Representativeness is dependent upon the proper design of the sampling program and will be satisfied by ensuring that the FSP is followed and the proper sampling techniques are used.

4.4.3 Measures to Ensure Representativeness of Laboratory Data

Representativeness in the laboratory is ensured by using the proper analytical procedures, meeting sample holding times, and analyzing and assessing field duplicated samples. The sampling network is designed to provide data representative of Site conditions. During the development of this sampling network, consideration was given to known past facility operations, existing analytical data, and site setting. The rationale for this project's sampling program is discussed in detail in Section 5 of the FSP.

4.5 COMPARABILITY

4.5.1 Definition

Comparability is an expression of the confidence with which one data set can be compared with another. Comparability is also dependent on similar QA objectives.

4.5.2 Measures to Ensure Comparability of Field Data

Comparability is dependent upon the proper design of the sampling program and will be satisfied by ensuring the FSP is followed and the proper field sampling techniques are used.

4.5.3 Measures to Ensure Comparability of Laboratory Data

Planned analytical data will be comparable when similar sampling and analytical methods are used and documented in the QAPP. Comparability is also dependent on similar QA objectives.

4.6 SENSITIVITY

Sensitivity is a calculated and measured response of a substance with 99 percent confidence that the analyte concentration determined from laboratory analysis is greater than zero. Sensitivity is typically expressed in the form of detection limits. The sensitivities required for organic analyses performed according to the current USEPA CLP Statement of Work OLM03.1 and for inorganic analyses performed according to USEPA CLP Statement of Work ILM04.0 will be the contract required quantitation limits (CRQLs). These CRQLs are presented in Appendix C. The method detection limits for the remaining laboratory parameters are summarized in Table 4.3. The project reporting limits for the remaining parameters are summarized in Table 4.4.

4.7 LEVEL OF QUALITY CONTROL EFFORT

Field blank, trip blank, method blank, field duplicate, SRM, and MS samples will be analyzed to assess the quality of the data resulting from the field sampling program. Field and trip blank samples consisting of deionized/distilled water will be submitted to the analytical laboratory to provide a means of assessing the quality of the data resulting from the field sampling program.

Field rinseate blank (field blank) samples are analyzed to determine whether decontamination procedures on re-usable field equipment have been sufficient to prevent cross-contamination of subsequently collected samples. One field blank sample will be collected for every 10 investigative groundwater samples collected during this field program. Field blanks will not be collected during soil and sediment sampling activities since field blank samples are not required by USEPA Region V for these media.

Trip blank samples are used to assess the potential for contamination of samples due to contaminant migration during sample shipment and storage. Trip blanks only pertain to VOC samples. Trip blanks are prepared in the actual sample containers (40-mL glass vials) prior to the sampling event and are kept with the investigative samples throughout the sampling event. They are then packaged for shipment with the investigative samples (and

TABLE 4.4
NON-CLP PARAMETERS PROJECT REPORTING LIMITS

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

METHOD	REPORTING LIMIT
TCLP Volatiles - 1311/8240	**(mg/L)**
Benzene	0.025
Methyl ethyl ketone	0.050
Carbon tetrachloride	0.025
Chlorobenzene	0.025
Chloroform	0.025
1,2-Dichloroethane	0.025
1,1-Dichloroethylene	0.07
Tetrachloroethylene	0.025
Trichloroethylene	0.025
Vinyl chloride	0.050
TCLP Metals - 1311/6010A/7470	**(mg/L)**
Silver	0.5
Arsenic	0.5
Barium	10
Cadmium	0.1
Chromium	0.5
Lead	0.5
Selenium	0.25
Mercury	0.002
General Chemistry	
pH - 9045A	no units
Total organic carbon - Walkley-Black	50 * mg/kg
Reactive cyanide - 7.3.3	200 mg/kg
Reactive sulfide - 7.3.4	200 mg/kg
Flashpoint	deg F
Sulfur	61.2 mg/kg

* Quanterra's routine reporting limit for TOC by the Walkley-Black method, as noted in SOP NC-WC-0018, is 100 mg/kg. A project specific reporting limit of 50 mg/kg has been established for this AlliedSignal project.

sometimes other samples) and sent to the laboratory for analysis. At no time after their preparation will the trip blank sample containers be opened before they reach the laboratory. There will be at least one trip blank included in each sample shipment container (cooler) containing groundwater VOC samples. Trip blanks will not accompany soil or sediment VOC samples since trip blank samples are not required by the USEPA Region V for these media.

Method blank samples are generated within the laboratory and are used to assess contamination resulting from laboratory procedures. Field duplicate/replicate samples are analyzed to check for sampling and analytical reproducibility. One field duplicate will be collected for every 10 investigative samples, per matrix.

Matrix spike samples provide information on the effect of the sample matrix on the digestion and measurement methodology. All MS samples are performed in duplicate, with the duplicate referred to as a matrix spike duplicate (MSD). Hereinafter, the pair will be referred to as MS/MSD samples. MS/MSD samples are designated for organic analyses. For inorganic analyses, spike/duplicate or MS/MSD analyses are performed at the same frequency as the MS/MSD. MS/MSD samples (and spike/duplicate samples) are investigative samples on which additional analyses are performed. Additional sample volume (double or triple volume) for soil and groundwater MS/MSD samples will be collected based upon the requirements of the laboratory. One MS/MSD (and spike/duplicate) sample will be collected/designated for every 20 investigative soil, groundwater, and sediment samples collected, per matrix. The various QC samples estimated for this sampling program are summarized in Table 4.1.

6.2.1 Sample Receipt

Samples shall be received and logged in at the Quanterra laboratory by the sample custodian or her designee (a trained associate). Upon sample receipt, the sample custodian shall, as appropriate:

- Wear appropriate personal protective equipment. At a minimum, this consists of gloves, a laboratory coat, safety glasses, and in some cases a respirator.
- Examine the shipping containers to verify that the custody seals are intact.
- Examine all sample containers for damage.
- Open shipping containers in adequately ventilated areas to ensure worker safety.
- Determine if the temperature required by the requested testing program has been maintained during shipment. Document the shipping container temperature on the cooler receipt form.
- Compare samples received against those listed on the COC.
- Verify that sample holding times have not been exceeded.
- Examine all sample records for accuracy and completeness.
- Determine sample pH (if required for the scheduled analysis) and record on the cooler receipt form.
- Sign and date the COC immediately (only after shipment is accepted) and attach the waybill/airbill.
- Note any problems associated with the samples on the cooler receipt form. If problems exist, immediately initiate a Condition Upon Receipt Report (CUR) and notify the laboratory project manager, who in turn will notify the Parsons ES project manager.
- Attach appropriate laboratory sample container labels with the laboratory identification number and test specified on the label.
- Place the samples in the proper laboratory storage area.

A CUR is generated by the sample custodian during the sample log-in process to document anomalies identified upon the receipt of samples in the laboratory. These anomalies are outside of laboratory control and do not require corrective actions to be taken within the laboratory. The Parsons ES project manager shall be notified by the Quanterra project manager or designee of all CURs generated for the project samples. The laboratory project manager is responsible for resolving with the client how to proceed with the

1. The temperature of the sample will be checked to determine whether they are the same.
2. The pH electrodes will be connected to the pH meter and the meter turned on.
3. The temperature setting will be set based upon the temperature of the buffer and the electrode placed first into the buffer solution.
4. After the reading has stabilized, the calibration knob will be adjusted to display the correct value.
5. The procedure stated in Steps 3 and 4 will be repeated for the second buffer solution.
6. The sample pH will then be measured by placing the electrode in the sample. The reading displayed on the meter will be recorded.
7. The electrode will then be removed from the sample and rinsed off with distilled water.
8. The pH meter will be recalibrated each time it is turned off and turned back on. It will also be calibrated after approximately every 4 hours of continuous operation or after every 10 groundwater samples are collected, whichever occurs first.

The execution of the calibration process will be documented in the field logbook along with the standard used and the sample pH values. Critical spare parts such as replacement batteries will be within a days' access if required. However, depending on the situation, an inoperable or malfunctioning pH meter will be replaced with a working unit.

Specific Conductance Meter Calibration

The conductivity cells of the specific conductance meter will be cleaned and checked against known conductivity standards before it is taken into the field. In the field, the instrument will be checked daily with approved traceable reference standards. The calibration process is summarized below.

1. The probe will be placed in the conductivity standard solution.
2. The temperature knob of the instrument will then be set to the temperature of the standard solution.
3. The instrument will be adjusted to the appropriate scale and set at the value for the calibration standard.

4. The electrode will be removed from the standard solution and rinsed with distilled water.
5. The temperature of the meter will then be set at the temperature of the distilled water that will be used for an equipment field blank. The conductivity of the distilled water will be measured.
6. If the conductivity of the field blank (distilled water) is high, the water will be discarded and a new blank sample obtained.

The specific conductance meter will be re-calibrated each time it is turned off and turned back on. It will also be calibrated after approximately every 4 hours of continuous operation or after every 10 groundwater samples are collected, whichever occurs first. All readings and calibrations will be recorded in the field logbook.

Thermometer Calibration

Temperature readings of groundwater samples will be taken in the field using a thermometer. Prior to each use, the thermometer will be thoroughly visually inspected to ensure that there is no mercury separation, or other form of damage. The precision of the thermometer will assessed based on the degree of concurrence between multiple consecutive readings of an aqueous matrix of constant temperature. If the error (discrepancy) between three or more readings exceeds ± 0.5 °C, the thermometer will be discarded and a new thermometer used.

MicroTIP® Calibration

The MicroTIP® must be calibrated in order to display concentration in units equivalent to ppm. First a supply of Zero Gas, which contains no ionizable gases or vapors, is used to set MicroTIP's zero point. Then, Span Gas, containing a known concentration of an ionizable gas or vapor, is used to set the sensitivity.

Usually clean outdoor air will be suitable as Zero Gas. If there is any doubt, use a commercial source of Zero Grad Gas and a second sampling bag. A supply of Span Gas of the desired compound and concentration must be obtained for calibration. Observe proper handling techniques for all gases.

SECTION 8
ANALYTICAL PROCEDURES

8.1 FIELD SCREENING ANALYTICAL PROCEDURES

The procedures for field measurements of pH, specific conductance, and temperature and for the use of the PID are described in the instrument SOPs.

8.2 LABORATORY ANALYSES

All soil, groundwater, and sediment sample chemical analyses and the evaluation of soil for BTU content will be performed by Quanterra. The Quanterra laboratory is located at 4101 Shuffel Drive, N.W., North Canton, Ohio 44720, telephone number (330) 497-9396. The analyses of soil, sediment, and groundwater samples for organic compounds (VOCs, SVOCs, PAHs, pesticides/PCBs) will be according to the USEPA CLP Statement of Work OLM03.1. The analysis of soil, sediment, and groundwater samples for inorganic compounds (8 total metals and cyanide) will be according to the USEPA CLP Statement of Work ILM04.0. The remaining parameter analyses will be in accordance with Quanterra's laboratory-specific analytical SOPs presented in Appendix B. Table 8.1 provides a summary of the analytical procedures that will be used by the laboratory to analyze the samples collected during this field program.

The physical parameters (odor, color, and lithology) associated with the sediment samples will be determined by the Parsons ES field geologist, based on visual and olfactory observations.

The remaining soil geotechnical parameter analyses will be performed by Hanson Engineers, Inc. located at 1525 South Sixth Street, Springfield, Illinois, 62703, telephone number (217) 788-2450.

Table 8.2 provided a summary of the methodologies that will be used during the geotechnical sample analysis.

TABLE 8.1
SUMMARY OF CHEMICAL ANALYTICAL PROCEDURES
QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Analyte(s)	Sample Matrix	Laboratory Procedures
TCL VOCs, SVOCs, PAHs, Pesticides/PCBs	Soil Sediment Groundwater	CLP SOW OLM03.1
TAL 8 Total Metals and Cyanide	Soil Sediment Groundwater	CLP SOW ILM04.0
TCLP 8 Metals	Soil	TCLP Leachate Procedure: SW 846 Method 1311 TCLP Extraction Procedure: SW 846 Method 3005A/3010A Mercury Analysis: SOP CORP-MT-0005, Rev. No.1 Other Metals Analysis: SOP CORP-MT-0001NC, No. 1.1
TCLP VOCs	Soil	TCLP Leachate Procedure: SW 846 Method 1311 VOC Analysis: SOP CORP-MS-0002 (Rev. 1)
Reactive Cyanide	Soil	SW 846 Method 7.3.3.2/9010 (SOP NC-WC-0033, Rev. 0)
Reactive Sulfide	Soil	SW 846 Method 7.3.4.2/9030 (SOP LM-WALN-1335, Rev. 1.0)
Flash Point (Closed Cup)	Soil	ASTM Method D93-80 SW 846 Method 1010, Updates I SOP LM-WALN-1151, Rev.0
Sulfur	Soil	General Bomb Method: ASTM Method D129-91 Sulfate Analysis: SOP STL-WC-0028 (Rev. 0)
TOC	Soil	Walkley-Black Method 29-3.5.2 (SOP NC-WC-0018)
pH	Soil	SW-846 Method 9040A/9045C and MCAWW Method 150.1 SOP NC-WC-0010

Notes:

TCL refers to the target compound list of organic parameters.

TAL refers to the target analyte list of inorganic parameters.

CLP SOW OLM03.1 refers to the USEPA CLP Statement of Work for Organic Analysis, Multi-Media, Multi-Concentration.

CLP SOW ILM04.0 refers to the USEPA CLP Statement of Work for Inorganic Analysis, Multi-Media, Multi-Concentration.

TCLP refers to Toxicity Characteristic Leaching Procedure analyses.

SW 846 refers to Test Methods for Evaluating Solid Waste, Physical/Chemical Methods, Third Edition, November 1986, and its updates.

SOP refers to the Quanterra laboratory-specific standard operating procedures presented in Appendix B.

ASTM refers to American Society of Testing Materials.

Walkley-Black refers to Methods of Soil Analysis, 1982, Second Edition, Method 29-3.5.2 Walkley-Black Procedure.

MCAWW refers to Methods for Chemical Analysis of Water and Wastes, EPA-600/4-79-020, March 1983, and subsequent revisions.

TABLE 8.2
SUMMARY OF GEOTECHNICAL ANALYTICAL PROCEDURES

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Parameter	Sample Matrix	Laboratory Procedures
Grain Size (with hydrometer)	Soil	ASTM Method D-422 & ASTM Method D-421
Bulk Density & Porosity	Soil	EM 1110-2-1906
Permeability	Granular Soils	ASTM Method D-2434
Permeability	Cohesive Soils	ASTM Method D-5084
Specific Gravity*	Soil	ASTM Method D-854
BTU Content	Soil	ASTM Method D-2015

Notes:

ASTM refers to American Society of Testing Materials.

EM refers to Laboratory Soils Testing Engineers Manual EM 1110-2-1906, Department of the Army, Office of the Chief of Engineers, Washington DC.

* Specific gravity measurements are required to calculate porosity.

The following subsections present a general description of the preparation and instrumental procedures to be used by Quanterra for this project. Section 7 contains a general description of Quanterra's instrument calibration procedures. Detailed, method-specific applications of the procedures are described in Quanterra associated laboratory analytical SOPs.

8.2.1 Metals Analyses

Two techniques, inductively coupled plasma (ICP) atomic emission spectroscopy and atomic absorption (AA) spectroscopy, will be employed to measure levels of specified metals in the samples. Sample digestion is required prior to most ICP and AA analyses.

8.2. 1.1 Metals Sample Preparation Procedures

The SW 846 Method 3005A, acid digestion of waters and groundwaters for total recoverable metals, is used to prepare aqueous samples (surface water and groundwater samples) for analysis by ICP, flame AA and GFAA analysis of antimony. For the analysis of dissolved metals the sample must be filtered at the time of collection. A 50 mL aliquot of the acidified sample is heated with 1 mL concentrated HNO_3 and 5 mL 1:1 HCl until the volume is reduced to 15 mL to 20 mL. Filtering may be performed if insoluble material is present. The final volume is adjusted to 50 mL. In the SW 846 Method 7060A protocol, the sample is processed similarly to SW 846 Method 3020A except that hydrogen peroxide is used in addition to nitric acid and the sample undergoes a less rigorous volume reduction (approximately 50 mLs).

8.2.1.2 Metals Instrumental Methods

SW 846 Method 6010A - ICP Procedures

Inductively coupled plasma atomic emission spectroscopy determines elements in solution. All matrixes including groundwater, surface water, aqueous samples, industrial wastes, soils, sludges, and sediments require digestion by Methods 3005A (water).

Method 6010A provides a simultaneous multi-element determination by ICP. Samples are nebulized and the resulting aerosol is transported to the plasma. Element-specific atomic line emission spectra are produced by radio-frequency inductively coupled plasma. The spectra are dispersed and the lines monitored by photomultiplier tubes. The background will be measured and the results corrected for background levels.

SW 846 Method 7470A - Mercury by CVAA

Mercury will be determined in water samples using SW 7470A. Method 7470A is cold-vapor atomic absorption procedures for determining the concentration of mercury. Sample preparation is specified in the method. Following dissolution, mercury in the sample is reduced to the elemental state, separated from solution and passed through a cell positioned in the light path of an atomic absorption spectrometer or mercury-specific analyzer.

8.2.2 Other Analyses

SW 846 Method 8240 - GC/MS Volatile Organics Analysis for TCLP VOC Analyses (Soil)

The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer which is used is used to provide both qualitative and quantitative information.

If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a purging chamber. It is then analyzed by purge and trap.

In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature (40°C for soils) and the volatile components are efficiently transferred from the

aqueous phase to the vapor phase. the vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

SW 846 Method 9045C and SW 846 Method 9040A - Soil and Water pH

The pH of water samples will be measured in the laboratory potentiometrically using a standard pH meter. The pH meter will be routinely calibrated in accordance with manufacturer's recommended procedures using buffered standards that are checked daily. Soil samples are mixed either with Type II water or with a calcium chloride solution, depending on whether the soil is calcareous or non-calcareous. The pH of the solution is then measured with a pH meter.

Walkley Black (W-B) Titration - Total Organic Carbon (Soil)

The determination of organic carbon in soil samples is based on the reduction of dichromate ($Cr_2O_7^{-2}$) ion by organic matter, wherein the unreduced ($Cr_2O_7^{-2}$) is measured by titration.

Method 300.0 - Analysis of Anions by Ion Chromatography - Sulfur (Soil)

A filtered aliquot of sample is injected into a stream of carbonate-bicarbonate eluant in an ion chromatographic system of a guard column, separator column, suppressor column and conductivity detector. The guard and separator columns are packed with low-capacity, strongly basic anion exchange resin. Ions are separated based on their affinity for the

exchange sites of the resin. The separated anions are directed onto a suppressor column that contains cation exchange resin in the hydrogen form. The suppressor column reduces the background conductivity of the eluant to a low or negligible level (weakly conductive carbonic acid) and converts the anions to their highly conductive acid forms. The separated anions in their acid form are measured by conductivity. They are identified on the basis of retention time as compared to known standards. Quantification is accomplished by measuring the area of the resultant peaks and comparing to a calibration curve generated from known standards.

If the native concentration of target analytes in the unspiked sample is high relative to the spiking concentration, the differences in the native concentration between the unspiked sample and the spiked sample may contribute a significant error to the precision and accuracy calculations making the accuracy and precision measures unrepresentative of the true method and matrix performance. For this reason, if the native concentration is four or more times the spiked concentration, the MS recoveries will not be calculated, the MSs will not be re-analyzed or re-prepared, and no further corrective action may be necessary. Finally if, in the judgment of the analyst, an analytical process error has occurred appropriate re-analysis or re-preparation steps are implemented. In all situations, the evaluation and corrective actions performed will be clearly and completely documented in the laboratory report case narrative.

For analyses using a MS and sample duplicate, the single MS sample is evaluated for accuracy and the sample and DU are assessed for precision. The assessment follows the same logic and reporting convention as described above.

For those analyses which do not allow MSs, an LCS and sample duplicate will be analyzed with each batch of samples. Batch control will be the same as that described for LCS. The "within-batch" precision is measured by calculating the RPD of any target analytes found in the primary and duplicate analysis of the sample if the found amount is greater than five times the SRL.

Laboratory Batch QC for Field, Equipment and Trip Blanks

Trip blank vials are sent with empty sample containers to the field and are shipped back to the laboratory with the filled field sample containers. Field blanks are created in the field (Refer to Section 4 of the QAPP). Trip and field blanks analyzed during this field program will be reagent (deionized) water, and will only be associated with and collected during groundwater sampling. Trip blanks will be analyzed for VOCs. Field blanks will be analyzed for the same parameters as the associated investigative groundwater samples.

Field and trip blanks associated with groundwater samples will be processed in the same manner as the associated field samples, since the matrixes are compatible. No Field and trip blanks associated with soil and sediment samples will be collected and submitted for analysis during this sampling program.

Any additional qualifiers used by the laboratory in reporting the data will be explained in the case narrative.

Any reports that are rejected as incomplete or in error will be returned to the laboratory for correction. Quanterra will submit a revised, corrected report within 2 weeks of notification of a rejected report.

10.4 Parsons ES Data Validation and Reporting

Upon receipt of the data package, the Parsons ES data manager will inspect all data packages for completeness. The completeness of the data will be evaluated by auditing the data package for:

- COC records
- Technical holding time
- Required analytical methods
- Reporting limits
- Reporting format (the required second column confirmation and any required GC/MS confirmations, sample IDs, analysis dates and times, sample volume, appropriate units, etc. as appropriate)
- Laboratory and field QC reporting forms (blanks, calibrations, laboratory control samples, duplicates, matrix spikes, surrogates, etc. as appropriate)
- Appropriate supporting raw data
- Case narrative
- Completeness of results

The data package submitted by Quanterra should be sufficient to conduct the main site risk assessment. Details of any missing, incomplete or incorrect parts of the data packages will be given to Quanterra via a data resubmittal form. The resubmitted data will be stamped "Resubmitted on [date]" and attached to the original data package. All persons receiving data packages will receive copies of the resubmitted data from the laboratory.

SECTION 12
PREVENTATIVE MAINTENANCE

12.1 FIELD EQUIPMENT/INSTRUMENTS

The field equipment for this project includes a PID, thermometer, pH meter, and conductivity meter. The specific preventative maintenance procedures to be followed for these field instruments are those recommended by the manufacturer. Field instruments will be checked and calibrated before they are shipped or carried to the field. In addition, these instruments will be checked and calibrated daily before use in the field. Calibration checks will be documented on the field logbook.

In the event of equipment failure, backup instruments, spare parts, and/or replacement equipment will be present on-site or available within a one-day shipment period, to minimize delays in the field schedule. Table 12.1 provides an example of the minimum preventative maintenance procedures that apply to the field equipment.

12.2 LABORATORY INSTRUMENTS

The primary objective of a preventive maintenance program is to help ensure the timely and effective completion of a measurement effort by minimizing the down time of crucial sampling and/or analytical equipment due to expected or unexpected component failure. In implementing this program, efforts are focused in three primary areas: maintenance responsibilities; maintenance schedules; and adequate inventory of critical spare parts and equipment. The discussion below provides an overview of preventative maintenance associated with each of these areas. Summaries of preventative maintenance for laboratory equipment are provided in Tables 12.2 through 12.8.

TABLE 12.7
SUMMARY OF PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT - ION CHROMATOGRAPH [1]

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

As Needed	Daily	Weekly	Monthly	Semi-annually
Clean micromembrane suppressor when decreases in sensitivity are observed.	Check plumbing/leaks.	Check pump heads for leaks.	Check all air and liquid lines for discoloration and crimping, if indicated.	Lubricate left hand piston.
Check fuses when power problems occur.	Check gases.	Check filter (inlet).	Check/change bed supports guard and analytical columns, if indicated.	Check conductivity cell.
Reactivate or change column when peak shape and resolution deteriorate, or when retention time shortening indicates that exchange sites have been deactivated.	Check pump pressure.			Check conductivity cell for calibration.
De-gas pump head when flow is erratic.	Check conductivity meter.			

(1) Manufacturer's instructions for each instrument provide a detailed discussion on the complete maintenance operational requirements.

TABLE 12.8
SUMMARY OF PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT - MISCELLANEOUS EQUIPMENT

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Sonicator [1]

Daily	As Needed
Daily when used: Inspect probe tips for inconsistencies (etching/pitting).	Replace probe tip.
	Disassemble and clean sonicator probe tips.
	Tune sonicator assembly.

Analytical/Top Loading Balances[1]

Daily	Annually
Daily when used: Calibrate with check weights.	Internal weight train serviced. Gears and electronics serviced.

Refrigerators/Walk-in Coolers[1]

Daily	As Needed
Temperatures checked and logged.	Refrigerant system and electronics serviced.

Ovens[1]

Daily	As Needed
Temperatures checked and logged.	Electronics serviced.

Specific Digital Ion Analyzer[1]

Daily	As Needed
Daily when used: Calibrate with check standards.	Electronics serviced.

[1] Manufacturer's instructions for each instrument provide a detailed discussion on the complete maintenance operational requirements.

TABLE 12.8 (continued)
SUMMARY OF PREVENTATIVE MAINTENANCE FOR LABORATORY EQUIPMENT - MISCELLANEOUS EQUIPMENT

QUALITY ASSURANCE PROJECT PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Spectrophotometer[1]

As Needed	Daily	Monthly	Annually
Dust the lamp and front of the front lens.	Check the zero %T adjustment.	Perform wavelength calibration at 530 nm.	Oil bearings.

pH Meter[1]

As Needed	Daily
Clean electrode.	Verify electrodes are properly connected and filled.
Refill reference electrode.	Make sure electrode is stored in buffer.

[1] Manufacturer's instructions for each instrument provide a detailed discussion on the complete maintenance operational requirements.

APPENDIX B

QUANTERRA LABORATORY ANALYTICAL STANDARD OPERATING PROCEDURES

APPENDIX B-1

TCLP LEACHATE PROCEDURE

QUANTERRA INCORPORATED

SOP CHANGE FORM

SOP NUMBER:NC-IP-0005

SOP TITLE:Toxicity Characteristic Leaching Procedure (TCLP)

SOP SECTION(S) AFFECTED BY CHANGE:11.3.4.1.1

REASON FOR ADDITION OR CHANGE:Error discovered during review by client

CHANGE EFFECTIVE FROM: (DATE):02/24/97

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY)
[Identify change or issue document change with italics or change bar.]

Section 11.3.4.1.1 (Determination of appropriate extraction fluid for unfilterable samples) should proceed to Section 11.3.*5* (Extraction procedure), not to Section 11.3.*7* (Equipment cleanup procedure).

SUBMITTED BY/DATE:Ruby Weber 2/24/97

*APPROVED BY:

Technical Reviewer Signature Date 2-25-97

Environmental Health & Safety Signature Date 2-26-97

QA Signature Date 2/27/97

Management Signature Date 2/26/97

Must be same signature authorities of ~~SOP being~~ revised.

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: TOXICITY CHARACTERISTIC LEACHATE PROCEDURE (TCLP)

(SUPERSEDES: REVISION 0 (09/01/95))

Prepared by: _____

Reviewed by: _____
Technology Specialist

Approved by: _____
Quality Assurance Manager

Approved by: _____
Environmental Health and Safety Coordinator

Approved by: _____
Laboratory Director

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. The (TCLP) leachate procedure is designed to determine the mobility of inorganic, semivolatile organic, and volatile organic compounds present in liquid, solid, and multiphasic wastes. It is based on SW846 Method 1311.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. Samples are characterized according to their percent total solids composition and the resulting pH from the preextraction evaluation. A representative sample is weighed and the appropriate extraction fluid added. This preparation is tumbled on a rotary extractor for sixteen to twenty hours, filtered, and preserved.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Separatory funnel may be used to separate emulsions by stirring with a glass rod.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazards are known:

5.3.1. The following materials are known to be **corrosive: Hydrochloric Acid, Nitric Acid, Sodium Hydroxide, Acetic Acid**

5.4. The chemicals used in this procedure may be toxic or carcinogenic. Therefore, each chemical compound should be treated as a potential health hazard. From this viewpoint, exposure to these chemicals should be reduced to the lowest possible level.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.7. Secure tumbler and extraction bottles before starting rotary agitation apparatus.

5.8. Shield should always be on vessel to protect against loose bolts.

5.9. Remove and load ZHE extraction vessels evenly from rotary agitation apparatus.

5.10. During sample rotation, pressure may build up inside the bottle, periodic venting of the bottle will relieve excess pressure.

5.11. When opening the release valve on the pressure filter, make sure that the opening is pointing away from you since liquid sprays from the opening.

5.12. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Rotary agitation apparatus: capable of end-over-end rotation at 30 (±2) rpm.

6.2. Zero headspace extraction (ZHE) vessel.

6.3. Stainless steel high pressure filtration device: capable of 0-50 psi.

6.4. Glass fiber filters: Pore size 0.6 to 0.8 µm, diameter size 9.0 cm to 15.0 cm, or equivalent.

6.5. VOA vials: 40 mL with Teflon® lined caps.

6.6. Air source: capable of pressurizing vessels up to 60 lbs.

6.7. Top loading balance: capable of accurately weighing ± 0.01 grams.

6.8. Syringe: Luer tip, 50 cc.

6.9. pH meter capable of measuring ± 0.05 units at 25°C and calibrants (4, 7, and 10)

6.10. Glass bottles and caps: 1 liter and 2 liter with Teflon® liners.

6.11. Plastic bottles and caps: 1 liter and 2 liter

6.12. Nitrogen source: high purity

6.13. Tedlar® bags

6.14. Foam packing: used to secure sample bottles in tumbler

6.15. Glass funnels

6.16. Beakers: 100 mL

6.17. Graduated cylinders: various sizes

6.18. Magnetic stir bars

6.19. Stir plate

6.20. Hot plate

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. HCl, hydrochloric acid, concentrated: reagent grade

7.1.2. Water: reagent grade

7.1.3. 1N hydrochloric acid: Dilute 83 mL of concentrated HCl to 1 liter with reagent water.

7.1.4. HNO_3 nitric acid, concentrated: reagent grade

7.1.5. NaOH sodium hydroxide, pellets: reagent grade

7.1.6. 1N sodium hydroxide: Dissolve 40 g of NaOH pellets in reagent water. Dilute to 1 liter with reagent water.

7.1.7. CH_3COOH glacial acetic acid: reagent grade

7.1.8. Extraction fluid

7.1.8.1. Extraction fluid #1: Add 46 mL of CH_3COOH plus 514.4 mL of 1N NaOH and dilute to 8 liters with reagent water. After mixing well, the pH must be 4.93 (± 0.05). Record pH.

7.1.8.2. Extraction fluid #2: Dilute 46 mL of CH_3COOH to eight liters with reagent water. After mixing well, the pH must be 2.88 (± 0.05). Record pH.

NOTE: If larger volumes are required, increase all volumes proportionally.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. All samples for TCLP are refrigerated upon receipt at 4°C ± 2°C and are not chemically preserved.

8.2. TCLP extracts for Metals analysis are preserved with nitric acid to a pH < 2 and stored in glass or plastic bottles at room temperature.

8.3. TCLP extracts for Organics and ZHE analysis are stored in glass bottles with Teflon® lined caps at 4°C ± 2°C.

8.4. Holding times categorized by days: from sample collection to TCLP filtration completion

8.4.1. Volatiles: 14 days

8.4.2. Semivolatiles: 14 days

8.4.3. Metals: 28 days for mercury and 180 days for all other metals

	From: Field Collection To: TCLP Extraction	From TCLP Extraction To: Preparation Extraction	From Preparative Extraction To: Determinative Analysis	Total Elapsed Time
Volatiles	14	NA	14	28
Semi-volatiles	14	7	40	61
Mercury	28	NA	28	56
Metals, except Mercury	180	NA	180	360

NA = Not Applicable

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. Semi-volatile Organic Compounds and Metals

9.2.2.1. A Buffered Method Blank consisting of the extraction fluid added to samples within the analytical batch must be extracted with each analytical batch of samples.

9.3. Spiking Solutions

9.3.1. Spiking Solutions (LCS, MS/MSD, Surrogates) are added where applicable according to the corresponding preparation or analytical SOP.

10. CALIBRATION AND STANDARDIZATION

10.1. Not Applicable

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation Procedure

11.3.1. Preliminary Extraction Procedures

11.3.1.1.If the sample is a liquid or a water, percent total solids needs to be determined.

11.3.1.1.1.If the liquid sample is an oil or a solvent, treat the sample as a solid (proceed to Section 11.3.4)

11.3.1.2.Perform these preliminary extraction evaluations and preparations (1) Determination of Total Solids and Filterability (Section 11.3.2). (2) Preparation for Filterable Samples (Section 11.3.3) (3) Determination of appropriate Extraction Fluid for Unfilterable Samples (Section 11.3.4). Samples should be allowed to warm to room temperature prior to extraction.

11.3.2. Determination of Total Solids (percentage) and Filterability

11.3.2.1.Dry a filter in the oven at 100°C ± 20°C for 10-15 minutes. Weigh 100 mL of the water sample into a beaker and record the weight. Weigh the dried filter and record its weight. Filter the sample using a vacuum filtration system. Remove the wet filter with accumulated solids and dry in the oven at 100° C ± 20°C for 10-15 minutes. Weigh the filter and record the weight. Determine and record % Total Solids in the TCLP or the ZHE logbook. See calculations below.

$$\%\ Total\ Solid = \frac{(dry\ filter + solid) - dry\ filter}{weight\ (g)\ of\ sample} \times 100$$

11.3.2.2.Filterability

11.3.2.2.1.If the percent Total Solid is < 0.5% for a semivolatile organics and/or Metals sample, the sample is filterable. (Proceed to Section 11.3.3.1)

11.3.2.2.2.If the percent Total Solid is <5.0% for a volatile organic sample (ZHE), the sample the sample is filterable (proceed to Section 11.3.3.2)

11.3.2.2.3. If the percent Total Solids is ≥ 0.5% for a semivolatile organics and/or metals sample, the sample is unfilterable (proceed to Section 11.3.4.1).

11.3.2.2.4. If the percent total solids is ≥ 5.0% for a volatile organic sample (ZHE), the sample is unfilterable (proceed to Section 11.3.4.2).

11.3.3. Preparation for Filterable Samples (samples that are < 0.5% Total Solid)

11.3.3.1. When preparing a semivolatile organics and/or metals filterable sample, the sample does not have to be tumbled. The sample is filtered into a glass or plastic bottle (Refer to Section 11.3.5.1.3). Take the pH of the filtrate with a pH meter and record on associated worksheet(s) and in the TCLP and/or ZHE logbook. Circle < 0.5% on the worksheet. The sample is accompanied by a filtered Buffer #1 Method Blank Sample. Extracts for semivolatile organic compounds are filtered into glass bottles with Teflon® lined caps and stored at 4°C ± 2°C. Extracts for metals analysis are preserved HNO_3 (pH < 2) and stored at room temperature in plastic or glass bottles. Deliver the bottled sample to the appropriate department(s) with the applicable worksheets.

11.3.3.2. When preparing a Volatile Organics (ZHE) filterable sample, the sample does not have to be tumbled, only filtered. To filter the sample, secure the glass inlet/outlet flange (bottom flange) onto the ZHE body in accordance with the manufacturer's instructions. Secure the glass fiber filter between the support screens and set aside. Set liquid inlet/outlet flange aside. Transfer aproximately 100 to 150 mL of sample quickly to the ZHE vessel. Secure the filter and support screens onto the top flange of the device and secure the top flange to the ZHE body in accordance with manufacturer's instruction. Tighten all ZHE fittings and place the device in the vertical position (gas inlet/outlet flange on the bottom). Attach a gas line to the gas inlet/outlet valve (bottom flange) and with the liquid inlet/outlet valve (top flange), begin applying gentle pressure of 1 psi to 10 psi (or more if necessary) to force all headspace slowly out of the ZHE device into a hood. At the first appearance of liquid from the liquid/outlet valve, quickly close the valve and discontinue pressure. Attach a syringe (Luer tip) to the inlet/outlet valve and open the valve. Begin applying gentle pressure to force the liquid into the syringe. Collect 45 mL. Close the inlet/outlet valve and remove the syringe. Transfer as much of the 45 mL filtrate as possible to a 40 mL VOA vial. Cap the vial and invert to check for air bubbles. If air bubbles exist remove cap and add more liquid

filtrate. Repeat procedure, filling one or two more vials as described above. Label vials with sample I.D. and deliver to the appropriate department(s) with the applicable worksheets.

11.3.4. Determination of appropriate Extraction Fluid for Unfilterable Samples (oil, solvent, or samples with ≥ 0.5% Total Solid).

11.3.4.1. Semivolatile Organic Compounds and/or Metals

11.3.4.1.1. Homogenize the sample with a tongue blade and place 5 g in a 100 mL beaker or equivalent. If possible, sample should be reduced to a particle size of 1mm or less for the pre-test. Pour 96.5 mL of Reagent Water into the beaker, add a magnetic stir bar, and place on a stir plate for approximately five minutes. Take and record the pH in the TCLP logbook. If the pH is ≤ 5, use Extraction Fluid #1 in the tumbling procedure. If the pH of the sample is > 5, add 3.5 mL of 1N HCl. Place on a hot plate (95°C ± 4°C) and heat for ten minutes. Allow the sample to cool. Take and record the pH in the TCLP logbook. If the pH is ≤ 5, use Extraction Fluid #1 in the tumbling procedure. If the pH is > 5, use Extraction Fluid #2. Record on the worksheet buffer used. Proceed to Section 11.3.5.

11.3.4.2. Volatile Organic Compounds (ZHE)

11.3.4.2.1. Extraction Fluid #1 is always used in the tumbling procedure. Proceed to Section 11.3.6.

11.3.5. Extraction Procedure for Semivolatile Organic Compounds and/or Metals

11.3.5.1. Extraction of Semivolatile Organic Compounds and/or Metals that will obviously yield no free liquid when subjected to pressure filtration.

11.3.5.1.1. Homogenize sample with a tongue blade and weigh 100 g of sample into an appropriate bottle (glass for organics and plastic or glass for metals). For a Semivolatile Organic and/or Metals extraction, sample should undergo particle size reduction to 1.0 cm.

11.3.5.1.2.Add 2 liters of appropriate Extraction Fluid as Determined in 11.3.4.1.1 Cap the bottle tightly and secure in the rotary agitation apparatus with foam packing. Record rotary agitation apparatus I.D. associated with each sample in the TCLP logbook. Tumble sample at 30 rpm ± 2 rpm for 16 to 20 hours.

NOTE: Ambient room temperature shall be maintained (23°C ± 2°C) during the extraction period.

11.3.5.1.3.Remove the bottle and allow solids to settle out. Filter the sample. Filtration is achieved by using a 60 psi air or nitrogen source to force the sample through a filter. The filtration device (filter holder) is cleaned and rinsed with Reagent Water before and after every use and between samples. The filtration device is stainless steel. Filter pores are 0.6 μm to 0.8 μm in size. Connect the pressure hose to the filtration device. Place a clean bottle (amber glass for organics and plastic or glass for metals), marked with the appropriate sample I.D. and place under the device. Place the outlet hose so it drains into the bottle. Slowly increase the air pressure to the filtration device by small increments until the desired volume of sample is collected.

11.3.5.1.4.Take the pH of the filtrate with a pH meter and record in the TCLP logbook and on appropriate worksheets. Cap the bottle. Wipe off any spillage from the sample extract bottle with a paper towel. Extracts for semivolatile organic compounds are stored at 4°C ± 2°C. Extracts for metals analysis are preserved with HNO_3 (pH < 2) and stored at room temperature. Deliver the extract to the appropriate department(s) with the applicable worksheet(s).

11.3.5.2.Extraction of Semivolatile Organic Compounds and/or Metals with ≥ 0.5% total Solid that will yield free liquid when subjected to pressure filtration.

11.3.5.2.1.A minimum of 100 g of waste material is filtered to generate the solids utilized in the extraction. The volume of extract needed to perform the requested tests must be considered.

11.3.5.2.2.Weigh the portion of sample to be filtered. Record weight in comment section of the logbook.

11.3.5.2.3.Label a collection bottle with the sample I.D.

11.3.5.2.4.Filter the sample through a stainless steel high pressure filtration device. Collect the filtrate in a glass bottle if the sample is needed for organics analysis. Plastic or glass bottles are used to store filtrates for metals analysis.

11.3.5.2.5.Store the filtrates at 4°C ± 2°C.

11.3.5.2.6.Release the pressure in the filtration apparatus and disassemble. Carefully place the filter paper and "solid" collected into appropriate labeled tumbling container (glass if organics, plastic or glass if metals).

11.3.5.2.7.Add the appropriate extraction fluid as determined in Section 11.3.4.1.1. The volume of extraction fluid needed is determined by using the following formula:

VOLUME = 20 x (% Solid/100) x Filtered Waste (g).

11.3.5.2.8.Tumble for 16 to 20 hours at a rate of 30 rpm ± 2 rpm.

NOTE: Ambient room temperature is maintained at 23° ± 2°C during the extraction period.

11.3.5.2.9.Filter according to Section 11.3.5.1.3.

11.3.5.2.10.To determine the compatibility of initial filtrate and tumbled filtrate: Place 5 mL of the appropriate extraction fluid (Refer to Section 11.3.4.1.1) and 5 mL of the initial filtrate in a 4 mL VOA vial. Mix well and examine for miscibility.

11.3.5.2.11.If the initial filtrate and the filtrate obtained from tumbling are compatible, combine the two solutions and mix well. This combined solution is defined as the TCLP extract.

11.3.5.2.12.If the initial filtrate and the tumbled filtrate are not compatible, they are to be prepared and analyzed separately and the results mathematically combined. Refer to Data Analysis and Calculation, Section 12.1.

11.3.6. Extraction Procedure for Volatile Organic Compounds

11.3.6.1. Extraction Procedure for Volatile Organic Compounds with ≥ 5.0% Total Solid that will yield no free liquid when subjected to pressure filtration.

11.3.6.1.1. Homogenize and weigh 15 g of the sample into the ZHE cylinder. Add 300 mL of extraction fluid #1 to the cylinder. Place the filter pad between the two screens of the top piece of the ZHE and tighten down. Write the sample number and vessel letter on the ZHE Logsheet. Pressurize the ZHE cylinder with nitrogen to 50 psi. Tumble for sixteen to twenty hours at a rate of 30 (± 2) rpm. After tumbling is complete, depressurize the ZHE cylinder and filter the sample into VOA vials or Tedlar® bags making sure there is zero headspace in the filled vial or bag. Deliver the sample extract to the appropriate department(s) with applicable worksheet(s). The sample extract is stored at 4°C ± 2° C.

11.3.6.2. Extraction of Volatile Organic Compounds with ≥ 5.0% Total Solids that will yield free liquid when subjected to pressure filtration.

11.3.6.2.1. Homogenize and weigh an appropriate size subsample of the waste into the ZHE and record the mass. Carefully place the glass fiber filter between the support screens and secure to the ZHE. Tighten all the fittings.

11.3.6.2.2. Place the ZHE in a vertical position; open both the gas AND liquid inlet/outlet valves. Attach a gas line to the gas inlet/outlet valve.

11.3.6.2.3. Carefully apply gentle pressure of 10 psi (or more, if necessary) to force all headspace slowly out of the ZHE. At the FIRST appearance of liquid from the liquid inlet/outlet valve, quickly close the valve and discontinue gas pressure.

11.3.6.2.4. Assemble a syringe and place the plunger in all the way. Adjust the tension on the plunger to provide slight drag. Attach the syringe to the liquid inlet/outlet valve and open the valve.

11.3.6.2.5.Carefully apply gas pressure of no more than 10 psi to force out the liquid phase. Allow the sample to filter until no SIGNIFICANT additional filtrate has passed in a 2 minute period. If the capacity of the syringe is reached, close the liquid inlet/outlet valve, discontinue gas pressure, remove the syringe and return to Section 11.3.6.2.4.

11.3.6.2.6.Repeat previous step increasing the pressure in 10 PSI increments until 50 psi is reached. Remove the syringe and record the total filtrate volume. Close the valve and discontinue gas pressure. Transfer the filtrate to VOA vials and label appropriately.

11.3.6.2.7.Based on the iformation from Section 11.3.2.1 determine the volume of Fluid to load into the ZHE on the "solid" phase, the ZHE can therefore accommodate a maximum of 25 grams of "solid".

11.3.6.2.8.Load the fluid transfer reservoir with an excess of Fluid #1 and preflush the transfer line to eliminate air pockets. Be sure the required volume remains.

11.3.6.2.9.Attach the transfer line to the liquid inlet/outlet valve and open the valve. Make sure the gas inlet/outlet valve is open. Carefully pump the required volume into the ZHE and close both valves. Disconnect the transfer line.

11.3.6.2.10.Check the ZHE to make sure all the valves are closed and manually rotate the ZHE (end-over-end) 2 0r 3 times. Reposition the ZHE in the vertical position.

11.3.6.2.11.Pressurize the ZHE to 5-10 psi and slowly bleed out any headspace. Re-pressurize to 5-10 psi, recheck the pressure.

11.3.6.2.12.Slowly open the liquid inlet/outlet valve to bleed out any headspace that may have been introduced during the introduction of the Fluid. Upon the first sign of liquid from the valve, close the valve.

11.3.6.2.13.Repressurize the ZHE to 5-10 psi and place in the rotary agitator. Rotate at 28-32 rpm for 16-20 hours. Room temperature should be 23 ± 2 degrees Centigrade. Record the tumbler speed (RPMs) on the benchsheet.

11.3.6.2.14.Confirm that the pressure of 5-10 psi was maintained throughout the leaching. If it was NOT maintained, return to Section 11.3.6.2.1 and repeat the leachate with a new aliquot of sample.

11.3.6.2.15.Attach a syringe and open the liquid inlet/outlet valve to collect the aqueous leachate.

11.3.6.2.16.If the waste contained an initial filtrate (Section 11.3.6.2.5) that is miscible with the solid phase leachate (as determined in Section 11.3.5.2.10), the solid phase leachate may be directly recombined in the correct proportions (see Section 12.1.2) with the initial filtrate. If the individual phases are NOT compatible, they are to be collected, prepped and analyzed separately.

11.3.6.2.17.Following collection, store the TCLP leachate in 3 20 ml VOA vials with minimal headspace at 4 ± 2°C and prepare for analysis as soon as possible using the appropriate organic extraction procedure.

11.3.6.2.18.If the individual phases are analyzed separately, combine the results mathematically by using the recombination calculation in Section 12.1.

11.3.7. Equipment Cleanup Procedure

11.3.7.1.Release the pressure and disassemble the filtration apparatus. Clean all the pieces thoroughly, inside and out, with a quality soap. It may be necessary to use strong cleaners, such as solvents or acids to remove all the sample from the filter device. Rinse all the pieces of the filtration apparatus with plenty of Reagent Water after the initial cleaning.

11.3.7.2.Release the pressure and disassemble the ZHE unit and clean all the pieces thoroughly, inside and out, with a quality soap. It may be necessary to use strong cleaners, such as solvents or acids to remove all the sample

from the filter device. Rinse all the pieces of the ZHE with plenty of Reagent Water after the initial cleaning.

11.4. Sample Analysis Procedure

11.4.1. Not Applicable (Refer to the Applicable Analytical SOPs).

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Mathematical recombination of analytical results:

$$\textit{Final Analyte concentration} = \frac{(V_1)(C_1) + (V_2)(C_2)}{V_1 + V_2}$$

Where:

V_1 = ***Total Volume of Initial Filtrate Phase (L),***

C_1 = ***Analyte Concentration in Initial Filtrate Phase (mL/L),***

V_2 = ***Volume of Second Phase (L),***

C_2 = ***Analyte Concentration in Second Phase (mL/L), and where V_2 = Solid Phase Mass x 20.***

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Acid waste is disposed of in the sample prep sink with a copious amount of water.

15.2. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan.

15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. EPA Federal Register, 40 CFR, Parts: 261, 264, 265, 268, 271, 302

16.1.2. SW846, Test Methods for Evaluating Solid Waste, Third Edition, Toxicity Characteristic Leachate Procedure, Method 1311.

16.2. Associated SOPs

16.2.1. Total Solids in Aqueous Samples, NC-WC-0004

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. Not Applicable

17.2. Troubleshooting guide

17.2.1. The ZHE should be checked for leaks after every extraction.

17.2.2. Contamination can arise from improper washing of the filtration apparatus or the ZHE vessel, impurities in the extraction fluid, and impurities in the air line. It is recommended that vessels which contained samples with results > RL be checked for contamination after they have been cleaned. Contamination checks can be run by tumbling a sample to see if it comes up clean, or a blank may be tumbled & checked for cleanliness.

APPENDIX B-4

ICP METHOD FOR TRACE ELEMENT ANALYSIS

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Quanterra
Environmental Services

QUANTERRA INCORPORATED

SOP NUMBER:CORP-MT-0001NC Revision 1.1

SOP TITLE: **INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7**

SOP SECTION(S) AFFECTED BY CHANGE:11.17

REASON FOR ADDITION OR CHANGE:Error discovered during review by client

CHANGE EFFECTIVE FROM: (DATE):02/24/97

CHANGE OR ADDITION (SPECIFY SECTION: USE ADDITIONAL SHEETS IF NECESSARY)
[Identify change or issue document change with italics or change bar.]

Section 11.17: "The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix *E* provides guidance on performing MSA analyses."...

...Reference should be Appendix *D* "MSA Guidance"

SUBMITTED BY DATE:Ruby Weber 2/24/97

APPROVED BY:

Technical Reviewer Signature Melissa Balt Date 2-25-97

Corporate QA or Technical Reviewer Signature [signature] Date 2/25/97

Environmental Health & Safety Signature [signature] Date 2-25-97

QA Signature [signature] Date 2/25/97

Management Signature Kevin Anthony Date 2/26/97

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: INDUCTIVELY COUPLED PLASMA-ATOMIC EMISSION SPECTROSCOPY, SPECTROMETRIC METHOD FOR TRACE ELEMENT ANALYSIS, METHOD 6010A AND METHOD 200.7

(SUPERSEDES: CORP-MT-0001 (REVISION 1))

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TABLE OF CONTENTS

LIST OF APPENDICES:

1. SCOPE AND APPLICATION

1.1. This procedure describes the analysis of certain metals by Inductively Coupled Plasma Atomic Emission Spectroscopy (ICP) using SW-846 protocol Method 6010A and Methods for Chemical Analysis of Waters and Wastes protocol, Method 200.7 as published in 40 CFR Part 136, Appendix C. Table I of Appendix A lists the elements approved for analysis by Methods 6010A and 200.7. Additional elements may be analyzed under Methods 6010A and 200.7 provided the method performance criteria presented in Section 13.0 are met.

1.2. ICP analysis provides for the determination of metal concentrations over several orders of magnitude. Detection limits, sensitivity and optimum concentration ranges of the metals will vary with the matrices and instrumentation used.

1.3. Method 6010A is applicable to the determination of dissolved, suspended, total recoverable and total elements in ground water, aqueous samples, soils, sludges, wastes, sediments, and TCLP, EP and other leachates/extracts. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples. Although digestion is not specifically required by the method, some clients and regulators do require digestion of dissolved samples and this must be clarified before project initiation.

1.4. Method 200.7 is applicable to the determination of dissolved, suspended, total recoverable and total elements in surface water, domestic and industrial waste waters. All matrices require digestion prior to analysis with the exception of analyses for dissolved metals in filtered and acidified aqueous samples if the criteria in Section 11.1 are met.

1.5. This SOP is not applicable to the analysis of drinking water samples due to the wide array of state specific requirements which must be accommodated. Refer to facility specific SOPs for guidance on performing drinking water analyses.

2. SUMMARY OF METHOD

2.1. This method describes a technique for the determination of elements in solution. The basis of the method is the measurement of atomic emission by an optical spectroscopic technique. Samples are nebulized and the aerosol that is produced is transported to the plasma torch where excitation occurs. Characteristic atomic-line emission spectra are produced by a radio frequency inductively coupled plasma (ICP). The spectra are dispersed by a grating spectrometer and the intensities of the lines are monitored by photomultiplier tubes. The photocurrents from the photomultiplier tubes are processed and controlled by a computer system. A background correction

technique is required to compensate for variable background contribution to the determination of trace elements. Background must be measured adjacent to analyte lines during analysis. The position selected for the background intensity measurement, on either or both sides of the analytical line, will be determined by the complexity of the spectrum adjacent to the analyte line. The position used must be free of spectral interferences and reflect the same change in background intensity as occurs at the analyte wavelength measured. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result. The possibility of additional interferences should also be recognized and appropriate actions taken.

2.2. Consult the appropriate SOP's for details on sample preparation methods.

3. DEFINITIONS

3.1. Dissolved Metals: Those elements which pass through a 0.45 um membrane. (Sample is acidified after filtration).

3.2. Suspended Metals: Those elements which are retained by a 0.45 um membrane.

3.3. Total Metals: The concentration determined on an unfiltered sample following vigorous digestion.

3.4. Total Recoverable Metals: The concentration determined on an unfiltered sample following treatment with hot, dilute mineral acid.

4. INTERFERENCES

4.1. Spectral, physical and chemical interference effects may contribute to inaccuracies in the determinations of trace elements by ICP. Spectral interferences are caused by:

- Overlap of a spectral line from another element.
- Unresolved overlap of molecular band spectra.
- Background contribution from continuous or recombination phenomena.
- Stray light from the line emission of high concentration elements.

4.1.1. A background correction technique is required to compensate for variable background contribution to the determination of trace elements. Background correction is not required in cases where a background corrective measurement would actually degrade the analytical result.

4.1.2. Interelement correction factors (IEC's) are necessary to compensate for spectral overlap. Interelement interferences occur when elements in the sample emit radiation at wavelengths so close to that of the analyte that they contribute significant intensity to the analyte channel. If such conditions exist, the intensity contributed by the matrix elements will cause an excessively high (or sometimes low) concentration to be reported for the analyte. Interelement corrections (IEC's) must be applied to the analyte to remove the effects of these unwanted emissions. To calculate an IEC, divide the observed concentration of the analyte by the observed concentration of the "interfering element."

4.1.3. Physical interferences are generally considered to be effects associated with sample transport, nebulization and conversion within the plasma. These interferences may result in differences between instrument responses for the sample and the calibration standards. Physical interferences may occur in the transfer of solution to the nebulizer (e.g., viscosity effects), at the point of aerosol formation and transport to the plasma (e.g., surface tension) or during excitation and ionization processes within the plasma itself. Changes in viscosity and surface tension can cause significant inaccuracies, especially in samples containing high dissolved solids or high acid concentrations. If physical interferences are present, dilution of the sample, use of a peristaltic pump, use of an internal standard and/or use of a high solids nebulizer can reduce the effect.

4.1.4. Chemical interferences are characterized by molecular compound formation, ionization effects and solute vaporization effects. Normally these effects are not significant with the ICP technique but if observed can be minimized by buffering the sample, matrix matching or standard addition procedures.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory.

5.3.1. The following materials are known to be **corrosive**:

sulfuric acid, hydrochloric acid, nitric acid and hydrofluoric acid. (NOTE: sulfuric acid is used in cleaning the ICP torch and hydrofluoric acid is commonly used in air toxics preparations.)

5.3.2. The following materials are known to be **oxidizing agents**:

nitric acid and hydrogen peroxide.

5.3.3. The plasma emits strong UV light and is harmful to vision. AVOID looking directly at the plasma.

5.3.4. The RF generator produces strong radio frequency waves, most of which are unshielded. People with pacemakers should not go near the instrument while in operation.

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Metals digestates can be processed outside of a fume hood. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.7. The use of hydrofluoric acid requires special safety precautions. Consult the facility EH&S Manager and laboratory supervisor for guidance.

6. EQUIPMENT AND SUPPLIES

6.1. Inductively Coupled Plasma Atomic Emission Spectrometer equipped with autosampler and background correction.

6.2. Radio Frequency Generator.

6.3. Nitrogen or argon gas supply, welding grade or equivalent.

6.4. Coolflow or appropriate water cooling device.

6.5. Peristaltic Pump.

6.6. Calibrated automatic pipettes or Class A glass volumetric pipettes.

6.7. Class A volumetric flasks.

6.8. Autosampler tubes.

7. REAGENTS AND STANDARDS

7.1. Intermediate standards are purchased as custom Quanterra multielement mixes or as single element solutions. All standards must be stored in FEP fluorocarbon or previously unused polyethylene or polypropylene bottles. Intermediate standard solutions must be replaced prior to the expiration date provided by the manufacturer. If no expiration date is provided, the intermediate solutions may be used for up to one year and must be replaced sooner if verification from an independent source indicates a problem.

7.2. Working calibration and calibration verification solutions may be used for up to 3 months and must be replaced sooner if verification from an independent source indicates a problem. Standards should be prepared in a matrix of 5% hydrochloric and 5% nitric acids. An exception to this is in the event the Trace ICP is utilized without the internal standard. In this case, the standard acid matrix must be matched to the final preparation matrix as listed in Section 11.10.

7.3. Refer to Tables III, IV, IVA, V and VI (Appendix A) for details regarding the working standard concentrations for calibration, calibration verification, interference correction and spiking solutions.

7.4. Concentrated nitric acid (HNO_3), trace metal grade or better.

7.5. Concentrated hydrochloric acid (HCl), trace metal grade or better.

7.6. Reagent water must be produced by a Millipore DI system or equivalent. Reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Sample holding times for metals are six months from time of collection to the time of analysis.

8.2. Aqueous samples are preserved with nitric acid to a pH of <2 and may be stored in either plastic or glass. If boron or silica are to be determined, plastic containers are preferred. Refrigeration is not required. Preservation must be verified prior to analysis.

8.3. Soil samples do not require preservation but must be stored at 4°C ± 2° until the time of analysis.

9. QUALITY CONTROL

Table VII (Appendix A) provides a summary of quality control requirements including type, frequency, acceptance criteria and corrective action.

9.1. Initial Demonstration of Capability

Prior to analysis of any analyte using either Method 200.7 or Method 6010A, the following requirements must be met.

9.1.1. Instrument Detection Limit (IDL) - The IDL for each analyte must be determined for each analyte wavelength used on each instrument. The IDL must be determined annually. If the instrument is adjusted in any way that may affect the IDL, the IDL for that instrument must be redetermined. The IDL shall be determined by multiplying by 3, the standard deviation obtained from the analysis of a standard solution (each analyte in reagent water) at a concentration 3x - 5x the previously determined IDL, with seven consecutive measurements. Each measurement must be performed as though it were a separate analytical sample (i.e., each measurement must be followed by a rinse and/or any other procedure performed between the analysis of separate samples). The result of the IDL determination must be below the Quanterra reporting limit. The CLP IDL procedure can be used for this method.

9.1.2. Method Detection Limit (MDL) - An MDL must be determined for each analyte/matrix prior to the analysis of any samples. The MDL is determined using seven replicates of reagent water, spiked with all the analytes of interest, that have been carried through the entire analytical procedure. MDL's must be redetermined on an annual basis in accordance with 40 CFR Part 136 Appendix B requirements as detailed in Quanterra QA Policy QA-005. The spike level must be between the calculated MDL and 10X the MDL to be valid. The result of the MDL determination must be below the Quanterra reporting limit.

9.1.3. Linear Range Verification (LR) - The linear range must be determined on an annual basis for each analyte wavelength used on each instrument. The standards used to define the linear range limit must be analyzed during a

routine analytical run. The determined concentration of the linear range standard must be within 5% of the true value. The linear range is the concentration above which results cannot be reported without dilution of the sample. If the instrument is adjusted in any way that may affect the LR's, the LR's must be redetermined.

9.1.4. Background Correction Points - To determine the appropriate location for off-line background correction when establishing methods, the user must scan the area on either side adjacent to the wavelength and record the apparent emission intensity from all other method analytes. This spectral information must be documented and kept on file. Background correction points must be set prior to determining IEC's. Refer to the facility specific instrument operation SOP and ICP instrument manual for specific procedures to be used in setting background correction points.

9.1.5. Interelement Corrections (IEC) - ICP interelement correction factors must be determined prior to the analysis of samples and annually thereafter. If the instrument is adjusted in any way that may affect the IEC's, the IEC's must be redetermined. When initially determining IEC's for an instrument, wavelength scans must be performed to ensure that solutions in use are free from contaminants. If an IEC varies significantly from the previously determined IEC then the possibility of contamination should be investigated. The purity of the IEC check solution can be verified by using a standard from a second source or an alternate method (i.e., GFAA or ICP-MS). Published wavelength tables (e.g. MIT tables, Inductively Coupled Plasma-Atomic Spectroscopy: Prominent Lines) can also be consulted to evaluate the validity of the IEC's. Refer to the facility specific instrument operation SOP and instrument manufacturer's recommendations for specific procedures to be used in setting IECs. An IEC must be established to compensate for any interelement interference which results in a false analyte signal greater than $\pm$ the RL as defined in Tables I, IA or II.

Note: Trace ICP IEC's are more sensitive to small changes in the plasma and instrument setup conditions. Adjustments in the IEC's will be required on a more frequent basis for the Trace as reflected by the ICSA response.

9.1.6. Rinse Time Determination - To determine the appropriate rinse time for a particular ICP system, the linear range verification standard (see 9.1.3) should be aspirated as a regular sample followed by the analysis of a series of rinse blanks. The length of time required to reduce the analyte signals to < RL will define the rinse time for a particular ICP system. For some analytes it may be impractical to set the rinse time based on the linear range standard result (i.e., analyte not typically detected in environmental samples at that level and an

excessive rinse time would be required at the linear range level). Rinse time studies can be conducted at additional concentration levels. These additional studies must be documented and kept on file if a concentration other than the linear range level is used to set the rinse time. The concentration levels used to establish the rinse time must be taken into consideration when reviewing the data.

9.2. Method Blank (MB) - One method blank must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit (exception: common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in associated samples, whichever is higher (sample result must be a minimum of 20x higher than the blank contamination level).

- If the analyte is a common laboratory contaminant (copper, iron, lead (Trace only) or zinc) the data may be reported with qualifiers if the concentration of the analyte in the method blank is less than two times the RL. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- Repreparation and reanalysis of all samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples (see exception noted above).

- If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

- If the above criteria are not met and reanalysis is not possible, then the sample data must be qualified. **This anomaly must be addressed in the project narrative and the client must be notified.**

- For dissolved metals samples which have not been digested, a CCB result is reported as the method blank. The CCB run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCB.

9.3. Laboratory Control Sample (LCS) - One aqueous LCS must be processed with each preparation batch. The LCS must contain all analytes of interest and must be carried through the entire analytical procedure. Aqueous LCS spike levels are provided in Table III (Appendix A). The LCS is used to monitor the accuracy of the analytical

process. On-going monitoring of the LCS results provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

- If any analyte is outside established control limits the system is out of control and corrective action must occur. Until in-house control limits are established, a control limit of 80 - 120% recovery must be applied.

- In the event that an MS/MSD analysis is not possible a Laboratory Control Sample Duplicate (LCSD) must be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- In the instance where the LCS recovery is greater than 120% and the sample results are < RL, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the case narrative.**

- Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

- For dissolved metals samples which have not been digested, a CCV result is reported as the LCS. The CCV run immediately prior to the start of the dissolved sample analyses must be used for this purpose. No more than 20 samples can be associated with one CCV.

9.4. Matrix Spike/Matrix Spike Duplicate (MS/MSD) - One MS/MSD pair must be processed for each preparation batch. A matrix spike (MS) is a field sample to which known concentrations of target analytes have been added. A matrix spike duplicate (MSD) is a second aliquot of the same sample (spiked identically as the MS) prepared and analyzed along with the sample and matrix spike. Some client specific data quality objectives (DQO's) may require the use of sample duplicates in place of or in addition to MS/MSD's. The MS/MSD results are used to determine the effect of a matrix on the precision and accuracy of the analytical process. Due to the potential variability of the matrix of each sample, these results may have immediate bearing only on the specific sample spiked. Samples identified as field blanks cannot be used for MS/MSD analysis. Spiking levels are provided in Tables III and VI (Appendix A).

- If any analyte recovery or RPD falls outside the acceptance range, the recovery of that analyte must be in control for the LCS. Control limits of 80 - 120 % recovery and 20% RPD must be applied to the MS/MSD. If the LCS recovery is within limits, then the laboratory operation is in control and the results may be accepted. If the recovery of the LCS is outside limits corrective action must be taken. Corrective action will include repreparation and reanalysis of the batch. MS/MSD results which fall outside the control limits must be addressed in the narrative.

- If the native analyte concentration in the MS/MSD exceeds 4x the spike level for that analyte, the recovery data are reported as NC (i.e., not calculated). If the reporting software does not have the ability to report NC then the actual recovery must be reported and narrated as follows: "Results outside of limits do not necessarily reflect poor method performance in the matrix due to high analyte concentrations in the sample relative to the spike level."

- If an MS/MSD is not possible due to limited sample volume then a laboratory control sample duplicate (LCSD) should be analyzed. The RPD of the LCS and LCSD must be compared to the matrix spike RPD limits.

- For dissolved metals samples which have not been digested, a MS/MSD must be performed per batch of up to 20 samples by spiking two aliquots of the sample at the levels specified in Table III (Appendix A).

9.5. Serial Dilution (L) - Serial dilution analysis is performed to determine whether significant physical or chemical interferences exist due to the sample matrix. One sample per preparation batch must be processed as a serial dilution sample. The serial dilution is performed by running a sample at a 4x dilution. Samples identified as field blanks cannot be used for serial dilution analyses. The results of the diluted sample, after correction for dilution, should agree within 10% of the original sample determination when the original sample concentration is greater than 40x the IDL. If the results are not within 10%, narrate the possibility of chemical or physical interference.

9.6. High Calibration Standard (HCAL) - At the beginning of the run, prior to the analysis of samples, the high standard must be rerun and recovered within 95 - 105%. If any analyte of interest falls outside the acceptance criteria corrective action must occur. The analysis should be terminated, the problem resolved and the instrument recalibrated. (See Section 11.11 or 11.12 for required run sequence.)

9.7. Initial Calibration Verification (ICV/ICB) - Calibration accuracy is verified by analyzing a second source standard (ICV). For analyses conducted under Method 200.7, the ICV result must fall within 5% of the true value for that solution. For Method 6010A, the ICV must fall within 10% of the true value for that solution. An ICB is analyzed immediately following the ICV to monitor low level accuracy and system cleanliness. The ICB result must fall within +/- the reporting limit (RL) from zero. If either the ICV or ICB fail to meet criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the calibration reverified. (See Section 11.11 or 11.12 for required run sequence).

9.8. Continuing Calibration Verification (CCV/CCB) - Calibration accuracy is monitored throughout the analytical run through the analysis of a known standard after every 10 samples. The CCV must be a mid-range standard made from a 2x dilution of the

calibration standard. The CCV result must fall within *10%* of the true value for that solution. A CCB is analyzed immediately following each CCV. (See Section 11.11 or 11.12 for required run sequence.) The CCB result must fall within +/- RL from zero. Sample results may only be reported when bracketed by valid ICV/CCV and ICB/CCB pairs. If a mid-run CCV or CCB fails, the analysis for the affected element must be terminated, the problem corrected, the instrument recalibrated, the calibration verified and the affected samples reanalyzed. (Refer to Section 11.13 for an illustration of the appropriate rerun sequence).

9.9. Interference Check Analysis (ICSA/ICSAB) - The validity of the interelement correction factors is demonstrated through the successful analysis of interference check solutions. The ICSA contains only interfering elements, the ICSAB contains analytes and interferents. Refer to Table V (Appendix A) for the details of ICSA and ICSAB composition. Custom Quanterra multielement ICS solutions must be used. All analytes must be spiked into the ICSAB solution, therefore, if a non-routine analyte is required then it must be manually spiked into the ICSAB using a certified ultra high purity single element solution or custom lab-specific mix. Elements known to be interferents on a required analyte must be included in the ICP run when that analyte is determined. Aluminum, iron, calcium and magnesium must always be included in all ICP runs.

9.9.1. The ICSA and ICSAB solutions must be run at the beginning and end of the run or every 8 hours, whichever is more frequent. (See Section 11.11 or 11.12 for required run sequence.)

9.9.2. The ICSAB results for the interferents must fall within 80 - 120% of the true value. If any ICSAB interferent result fails criteria, the analysis should be terminated, the problem corrected, the instrument recalibrated and the samples rerun.

9.9.3. The ICSAB analytes must be recovered within 80 - 120% of the true value. If the ICSAB analytes do not meet criteria the analysis must be terminated, the problem corrected, the instrument recalibrated and the samples rerun for the affected analytes.

9.9.4. ICSA results for the non-interfering elements with reporting limits ≤ 10 ug/L must fall within the Quanterra guidelines of +/- 2x RL from zero. If the ICSA results for the non-interfering elements do not fall within +/- 2x RL from zero the field sample data must be evaluated as follows:

- If the non-interfering element concentration in the ICSA is the result of contamination versus a spectral interference, and this reason is documented, the field sample data can be accepted.

- If the affected element was not required then the sample data can be accepted.

- If the interfering elements are not present in the field sample at a concentration which would result in a false positive or negative result greater than +/- 2x RL from zero then the field sample data can be accepted.

- If the interfering element is present in the field sample at a level which would result in a false analyte signal greater than ± 2x RL from zero, the data can be accepted only if the concentration of the affected analyte in the field sample is more than 10X the analyte signal in the ICSA.

- If the data does not meet the above conditions then the IEC's must be re-evaluated and corrected if necessary and the affected samples reanalyzed or the sample results manually corrected through application of the new IEC to the raw results. If the results are recalculated manually the calculations must be clearly documented on the raw data.

9.10. CRI - To verify linearity near the RL for ICP analysis, a CRI standard is run at the beginning of each sample analysis run following the ICB. The CRI standard must contain all analytes at two times the RL. The CRI results must fall within 50% of the true value or the run must be terminated, the problem corrected and the instrument recalibrated.

9.11. Method of Standard Addition (MSA) -This technique involves adding known amounts of standard to one or more aliquots of the processed sample solution. This technique compensates for a sample interferent that may enhance or depress the analyte signal, thus producing a different slope from that of the calibration standards. It will not correct for additive interferences which cause a baseline shift. Refer to Section 11.17 for additional information on when MSA is required as well as Appendix D for specific MSA requirements.

9.12. Quality Assurance Summaries - Certain clients may require specific project or program QC which may supersede the SOP requirements. Quality Assurance Summaries (QAS) should be developed to address these requirements.

10. CALIBRATION AND STANDARDIZATION

10.1. Set up the instrument with the operating parameters recommended by the manufacturer. Allow the instrument to become thermally stable before beginning calibration (approximately 30 minutes of warm-up is required).

10.2. Calibrate the instrument according to the instrument manufacturer's recommendations. Refer to the facility specific instrument SOP and ICP instrument manual for detailed set up and operation protocols.

10.3. Calibration must be performed daily and each time the instrument is set up. Instrument runs may be continued over periods exceeding 24 hours as long as all calibration verification and interference check QC criteria are met. The instrument standardization date and time must be included in the raw data.

10.4. Refer to Section 9.0 for calibration verification procedures, acceptance criteria and corresponding corrective actions.

11. PROCEDURE

11.1. For 200.7 analyses, dissolved samples must be digested unless it can be documented that the sample meets all of the following criteria:

A. COD is < 20 ppm.
B. Visibly transparent with a turbidity measurement of 1 NTU or less.
C. Colorless with no perceptible order.
D. Is of one liquid phase and free of particulate or suspended matter following acidification.

11.2. A minimum of two exposures for each standard, field sample and QC sample is required. The average of the exposures is reported. For Trace ICP analyses, the results of the sum channel must be used for reporting.

11.3. Prior to calibration and between each sample/standard the system is rinsed with the calibration blank solution. The minimum rinse time between analytical samples is 60 seconds unless following the protocol outlined in 9.1.6 it can be demonstrated that a shorter rinse time may be used. Triton-X can be added to the rinse solution to facilitate the rinse process.

11.4. The use of an autosampler for all runs is strongly recommended.

11.5. The use of automated QC checks through the instrument software is highly recommended for all calibration verification samples (ICV,CCV), blanks (ICB,CCB,PB), interference checks (ICSA,ICSAB) and field samples (linear range) to improve the data review process.

11.6. To facilitate the early identification of QC failures and samples requiring rerun it is strongly recommended that sample data be reviewed periodically throughout the run.

11.7. To facilitate the data review and reporting processes it is strongly recommended that all necessary dilutions and post digestion spikes be performed before closing out the instrument run.

11.8. For unattended overnight auto-runs it is strongly recommended that the frequency of ICSA/ICSAB analysis be increased to every 4 hours.

11.9. The use of an internal standard is recommended on the non-Trace ICP's.

11.10. The use of an internal standard is required on the Trace ICP unless the calibration and QC standards are matrix matched to each digestion procedure used as follows:

Preparation Method	% HNO_3	% HCl
CLP Aqueous	1	5
CLP Soil	5	2.5
SW846 3050	5	5
SW846 3005	2	5
SW846 3010	3	5

The following procedural guidelines must be followed when using an internal standard:

11.10.1. Recommended internal standards are yttrium or scandium. (Note: Any element can be used that is not typically found in environmental samples at a high rate of occurrence.)

11.10.2. The internal standard (IS) must be added to every sample and standard at the same concentration. It is recommended that the IS be added to each analytical sample automatically through use of a third pump channel and mixing coil. Internal standards should be added to blanks, samples and standards in a like manner, so that dilution effects resulting from the addition may be disregarded.

11.10.3. The concentration of the internal standard should be sufficiently high to obtain good precision in the measurement of the IS analyte used for data correction and to minimize the possibility of correction errors if the IS analyte is naturally present in the sample.

11.10.4. The internal standard raw intensity counts must be printed on the raw data.

11.10.5. The analyst must monitor the response of the internal standard throughout the sample analysis run. This information is used to detect potential problems and identify possible background contributions from the sample (i.e., natural occurrence of IS analyte).

11.10.5.1. If the internal standard counts fall within 30% of the counts observed in the ICB then the data is acceptable.

11.10.5.2. If the internal standard counts in the field samples are more than 30% higher than the expected level, the field samples must then be screened without the addition of the internal standard.

11.10.5.3. If the internal standard element is not identified in the unspiked field sample at a level exceeding 10% of the level spiked, the data may be accepted.

11.10.5.4. If the IS analyte is detected in the unspiked field sample at a concentration greater than 10% of the spiked level then either:

- A different internal standard must be used.
- The IS concentration must be raised.
- The sample must be diluted and rerun.
- The analysis must be run without an internal standard (matrix matching must be substituted.)

11.11. The following analytical sequence must be used for Methods 6010A and 200.7:

Instrument Calibration
ICV
ICB
CRI
HCAL
ICSA
ICSAB
6 samples
CCV
CCB
10 samples
CCV
CCB

Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB
ICSA
ICSAB
CCV
CCB

Refer to Quality Control Section 9.0 and Table VII (Appendix A) for Method 6010A and 200.7 quality control criteria.

11.12. The following run sequence is consistent with 200.7, 6010A and CLP requirements and may be used as an alternate to the sequence specified in 11.11 if multiple methods must be accommodated in the same analytical run:

Instrument Calibration
ICV
ICB
CRI
HCAL
ICSA
ICSAB
CCV
CCB
10 samples
CCV
CCB
10 samples
CCV
CCB
Repeat sequence of up to 10 samples between CCV/CCB pairs as required to complete run
CCV
CCB
CRI
ICSA
ICSAB
CCV
CCB

Refer to the CLP SOP (CORP-MT-0002) for additional quality control requirements.

11.13. The following run sequence provides an illustration of a mid-run CCV or CCB failure and the appropriate corrective action run sequence as described in Section 9.8:

Original Run: Instrument Calibration
ICV
ICB
CRI
HCAL
ICSA
ICSAB
6 samples
CCV1
CCB1
10 samples
CCV2
CCB2
10 samples **
CCV3 *
CCB3 *
10 samples **
CCV4
CCB4
10 samples
CCV5
CCB5
ICSA
ICSAB
CCV6
CCB6

* Failure occurs at CCV3/CCB3
**Samples requiring rerun for affected analytes

Reanalysis: Recalibrate
ICV
ICB
CRI
HCAL
CCV2
CCB2
10 samples
CCV3
CCB3
10 samples
CCV4
CCB4
ICSA
ICSAB

CCV6
CCB6

Notes: If the reanalysis is conducted under the same instrument setup conditions then it is not necessary to rerun the ICSA/ICSAB at the start of the reanalysis sequence as long as the 8 hour criteria are met. If reanalysis can't be initiated immediately or under the same run conditions then reanalysis must be conducted using the full analysis sequence as detailed in Section 11.11.

Samples between CCV4 and CCV5 do not require reanalysis as they were bracketed by compliant QC samples.

See CORP-MT-0002 for the appropriate reanalysis sequence if CLP requirements must also be met.

11.14. Full method required QC must be available for each wavelength used in determining reported analyte results.

11.15. Guidelines are provided in the appendices on procedures to minimize contamination of samples and standards, preventive maintenance and troubleshooting.

11.16. All measurements must fall within the defined linear range where spectral interference correction factors are valid. Dilute and reanalyze all samples for required analytes that exceed the linear range or use an alternate wavelength for which QC data is established. If an interelement correction exists for an analyte which exceeds the linear range, the IEC may be inaccurately applied. Therefore, even if an overrange analyte may not be required to be reported for a sample, if that analyte is a interferent for any requested analyte in that sample, the sample must be diluted. Acid strength must be maintained in the dilution of samples.

11.17. For TCLP samples, full four point MSA will be required if all of the following conditions are met:

1) recovery of the analyte in the matrix spike is not at least 50%,

2) the concentration of the analyte does not exceed the regulatory level, and,

3) the concentration of the analyte is within 20% of the regulatory level.

The reporting and regulatory limits for TCLP analyses as well as matrix spike levels are detailed in Table VI (Appendix A). Appendix E provides guidance on performing MSA analyses.

11.18. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix,

radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.19. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

12. DATA ANALYSIS AND CALCULATIONS

12.1. ICV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(ICV)}{True(ICV)}\right)$$

12.2. CCV percent recoveries are calculated according to the equation:

$$\%R = 100\left(\frac{Found(CCV)}{True(CCV)}\right)$$

12.3. Matrix Spike Recoveries are calculated according to the following equation:

$$\%R = 100\left(\frac{SSR - SR}{SA}\right)$$

Where:
SSR = Spike Sample Result
SR = Sample Result
SA = Spike Added

12.4. The relative percent difference (RPD) of matrix spike/matrix spike duplicates or sample duplicates are calculated according to the following equations:

$$RPD = 100\left[\frac{|MSD - MS|}{\left(\frac{MSD + MS}{2}\right)}\right]$$

Where:
MS = determined spiked sample concentration
MSD = determined matrix spike duplicate concentration

$$RPD = 100\left[\frac{|DU1 - DU2|}{\left(\frac{DU1 + DU2}{2}\right)}\right]$$

Where:
DU1 = Sample result
DU2 = Sample duplicate result

12.5. The final concentration for a digested aqueous sample is calculated as follows:

$$mg/L = \frac{C \times V1 \times D}{V2}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V1 = Final volume in liters after sample preparation
V2 = Initial volume of sample digested in liters

12.6. The final concentration determined in digested solid samples when reported on a dry weight basis is calculated as follows:

$$mg/Kg, dry\ weight = \frac{C \times V \times D}{W \times S}$$

Where:
C = Concentration (mg/L) from instrument readout
D = Instrument dilution factor
V = Final volume in liters after sample preparation
W = Weight in Kg of wet sample digested
S = Percent solids/100

Note: A Percent Solids determination must be performed on a separate aliquot when dry weight concentrations are to be reported. If the results are to be reported on wet weight basis the "S" factor should be omitted from the above equation.

12.7. The LCS percent recovery is calculated according to the following equation:

$$\%R = 100\left(\frac{Found(LCS)}{True(LCS)}\right)$$

12.8. The serial dilution percent difference for each component is calculated as follows:

$$\%\,Difference = \frac{|I - S|}{I} \times 100$$

Where:
I = Sample result (Instrument reading)
S = Serial dilution result (Instrument reading × 4)

12.9. Appropriate factors must be applied to sample values if dilutions are performed.

12.10. Sample results should be reported with up to three significant figures in accordance with the Quanterra significant figure policy.

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file for each analyte of interest as described in Section 9.0.

13.2. Refer to Tables I, IA & II in Appendix A for the list of Method 6010A and 200.7 analytes as well as additional analytes that may be analyzed using this SOP.

13.3. Method performance is determined by the analysis of matrix spike and matrix spike duplicate samples as well as method blanks and laboratory control samples. The matrix spike recovery should fall within +/- 20 % and the matrix spike duplicates should compare within 20% RPD. Method blanks must meet the criteria specified in Section 9.2. The laboratory control samples should recover within 20% of the true value until in house control limits are established.

13.4. Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Environmental Health and Safety Director should be contacted if additional information is required.

15.2. Standards should be purchased and prepared in volumes consistent with laboratory use to minimize the volume of expired standards to be disposed.

16. REFERENCES

16.1. *Methods for the Determination of Metals in Environmental Samples, EPA/600/R-94/111, Supplement I, Revision 4.4, May 1994. Method 200.7.*

16.2. 40 CFR Part 136, Table IB, 7-1-92.

16.3. Test Methods for Evaluating Solid Waste , Physical/Chemical Methods, SW-846, 3rd Edition, Final Update I, July 1992. Method 6010A.

16.4. CORP-MT-0002, Inductively Coupled Plasma-Atomic Emission Spectroscopy, Method 200.7 CLP-M, SOW ILMO3.0.

16.5. QA-003, Quanterra QC Program.

16.6. QA-004, Rounding and Significant Figures.

16.7. QA-005, Method Detection Limits.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC.)

17.1. Modifications/Interpretations from reference method

17.1.1. Modifications from both Method 6010A and 200.7.

17.1.1.1. Method 200.7 and Chapter 1 of SW846 specify the use of reagent water with a purity equivalent to ASTM Type II water. This SOP requires that reagent water must be free of the analytes of interest as demonstrated through the analysis of method blanks.

17.1.1.2. The alternate run sequence presented in Section 11.12 is consistent with method requirements. Additional QC analyses were added to accommodate the CLP protocol requirements.

17.1.2. Modifications from Method 200.7.

17.1.2.1. Blank subtraction is not performed as per Quanterra QC policy. Method blank results are provided in the analytical report.

17.1.2.2. Method 200.7 defines the IDL as the concentration equivalent to a signal, due to the analyte, which is equal to three times the standard deviation of a series of ten replicate measurements of a reagent blank signal. Quanterra labs utilize the CLP IDL definition as defined in Section 9.1.1 of this SOP.

17.1.2.3. The calibration blank is prepared in an acid matrix of 5% HNO_3/5% HCl instead of the specified 2% HNO_3/10% HCl matrix as the former matrix provides for improved performance relative to the wide variety of digestate acid matrices which result from the various EPA preparation protocols applied.

17.1.2.4. Section 7.6.3 of 200.7 indicates that the QCS (ICV) should be prepared at a concentration near 1 ppm. The ICV specified in this SOP accommodates the 1 ppm criteria for the majority of analytes. For the remaining analytes, this SOP specifies ICV concentrations which are appropriate to the range of calibration. The intent of the ICV, verification of calibration standard accuracy, is independent of the ICV concentration used.

17.1.2.5. The ICS criteria applied by this SOP differ from those stated in the method. 200.7 states that results should fall within the established control limits of 1.5 times the standard deviation of the mean value. These control criteria were based on a specific solution made available by EMSL-Cincinnati to labs several years ago. Since this solution is no longer available, Quanterra has modeled their ICSA/ICSAB solutions on the design of the ICSA/ICSAB solution provided by EPA directly to EPA contract laboratories. The control limits listed in this SOP are those which EPA states applicable to the EPA designed solution.

17.1.2.6. Method 200.7 states the CCB should be within 2x the standard deviation of the average blank reading. The intent of this requirement is to ensure that the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

17.1.3. Modifications from Method 6010A.

17.1.3.1. Chapter 1 of SW-846 states that the method blank should not contain any analyte of interest at or above the MDL. This SOP states that the method blank must not contain any analyte of interest at or above the reporting limit. Common lab contaminants are allowed up to two times the reporting limit in the blank following consultation with the client.

17.1.3.2. Calibration is performed according to instrument manufacturer's recommendations. Method 6010A provides contradictory instructions for instrument calibration by stating to calibrate both according to instrument manufacturer's recommendations as well as to use a blank and three standards. TJA has stated that the use of multiple standards may be detrimental to determinations near the detection limit due to the inability of the linear equation to force-fit through the origin (see Appendix C) and recommends the calibration be performed using a blank and one standard. Leeman recommends a blank and three standards. EPA has stated that manufacturer's recommendations should take precedence and that the next version of the method, 6010B, will clear up the issue by stating "The calibration curve should consist of a minimum of a blank and a standard." This SOP requires verification of the initial instrument calibration using a CRI at two times the RL, an ICV at 5 - 25% of the calibration level, a CCV at 50% of the calibration level and by rerunning the high calibration standard (HCAL) post calibration to demonstrate linearity (See Tables I, IA and II).

17.1.3.3. Section 5.6 of 6010A states that the instrument check standard (CCV) should be prepared from a second source standard. This SOP states that the CCV will be from the same source as the calibration standards. The purpose of the second source standard is to verify the accuracy of the calibration standards. The intent of this requirement is met through the analysis of a second source ICV standard prior to the analysis of samples. The use of a same source CCV provides for a more accurate and consistent measure of instrument drift from initial calibration.

17.1.3.4. Section 5.7 states that spiking of the ICS solution with analytes is not required if the ICP will display overcorrection as a negative number. All Quanterra instrumentation has this capability and therefore the spike analysis is not required. Quanterra does run a spiked ICSAB but the analytes are not spiked at the 10x IDL level referenced in 6010A. The ICSAB solution run by Quanterra is based

on the design of the ICSA/ICSAB solution provided by EPA directly to contract environmental labs.

17.1.3.5. Method 6010A uses a Quality Control Standard (QCS) on a weekly basis to verify calibration standard accuracy. Quanterra refers to the QCS as an ICV and the accuracy verification is performed on a daily basis. The QCS described in Method 6010A is made to contain analytes at 10x the IDL. The Quanterra ICV solution is not made at 10x IDL for all elements as this concentration is not appropriate relative to the standard reporting limits. Quanterra designed the ICV to be a reliable indicator of calibration standard accuracy by raising the analyte concentrations to a level where the analytical determination is not impacted by low level curve bias.

17.1.3.6. Method 6010A states the CCB should be within 3x the standard deviations of the average blank reading. The intent of this requirement is to ensure that the calibration is not drifting at the low end. In the absence of guidance on how to determine the mean blank level from EPA, Quanterra has adopted an absolute control limit of +/- RL from zero for calibration blank criteria.

17.2. Modifications from previous SOP

None.

17.3. Facility Specific SOP's

Each facility shall attach a list of facility specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none. Refer to the appendices for any facility specific information required to support this SOP.

17.3.1. Refer to the SOP change form on file in North Canton's Quality Assurance department.

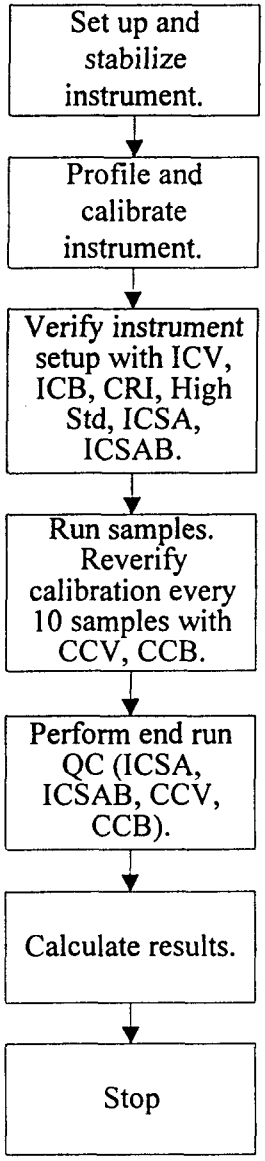
17.4. Documentation and Record Management

The following documentation comprises a complete ICP raw data package:

- Raw data (direct instrument printout signed by analyst).
- Relevant sample preparation benchsheets.

- Run log printout from instrument software where this option is available (TJA) or manually generated run log (i.e., Ward WSL printout).
- Data review checklist - See Appendix B.
- Standards documentation (including prep date, source and lot #).
- Non-conformance summary (if applicable).

17.5. Flow Diagram



APPENDIX A

TABLES

TABLE I. Method 200.7 and 6010A Analyte List

ELEMENT	Symbol	CAS #	6010A analyte	200.7 analyte	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Aluminum	Al	7429-90-5	X	X	200	20
Antimony	Sb	7440-36-0	X	X	60	6
Arsenic	As	7440-38-2	X	X	300	30
Barium	Ba	7440-39-3	X	X	200	20
Beryllium	Be	7440-41-7	X	X	5.0	0.5
Boron	B	7440-42-8		X	200	20
Cadmium	Cd	7440-43-9	X	X	5.0	0.5
Calcium	Ca	7440-70-2	X	X	5000	500
Chromium	Cr	7440-47-3	X	X	10	1
Cobalt	Co	7440-48-4	X	X	50	5
Copper	Cu	7440-50-8	X	X	25	2.5
Iron	Fe	7439-89-6	X	X	100	10
Lead	Pb	7439-92-1	X	X	100	10
Lithium	Li	7439-93-1	X		50	5
Magnesium	Mg	7439-95-4	X	X	5000	500
Manganese	Mn	7439-96-5	X	X	15	1.5
Molybdenum	Mo	7439-98-7	X	X	40	4
Nickel	Ni	7440-02-0	X	X	40	4
Phosphorus	P	7723-14-0	X		300	30
Potassium	K	7440-09-7	X	X	5000	500
Selenium	Se	7782-49-2	X	X	250	25
Silicon	Si	7631-86-9		X	500	50
Silver	Ag	7440-22-4	X	X	10	1
Sodium	Na	7440-23-5	X	X	5000	500
Strontium	Sr	7440-28-0	X		50	5
Thallium	Tl	7440-28-0	X	X	2000	200
Vanadium	V	7440-62-2	X	X	50	5
Zinc	Zn	7440-66-6	X	X	20	2

TABLE IA. Method 200.7 and 6010A Trace ICP Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Arsenic	As	7440-38-2	10	1.0
Lead	Pb	7439-92-1	3.0	0.3
Selenium	Se	7782-49-2	5.0	0.5
Thallium	Tl	7440-28-0	10	1.0
Antimony	Sb	7440-36-0	10	1.0
Cadmium	Cd	7440-43-9	2.0	0.2
Silver	Ag	7440-22-4	5.0	0.5
Chromium	Cr	7440-47-3	5.0	0.5

TABLE II. Non-Routine Analyte List

ELEMENT	Symbol	CAS #	Reporting Limit (ug/L) Water	Reporting Limit (mg/kg) Soil
Tin	Sn	7440-31-5	100	10
Titanium	Ti	7440-03-26	50	5
Bismuth	Bi	7440-06-99	200	20
Zirconium	Zr	7440-06-77	100	10
Tungsten	W	7440-03-37	500	50
Tellurium	Te	1349-48-09	500	50
Thorium	Th	7440-02-91	500	50
Uranium	U	7440-06-11	500	50
Palladium	Pd	7440-00-53	100	10

NOTE: Analysis of all elements listed may not be available at all Quanterra facilities.

TABLE III. Matrix Spike and Aqueous Laboratory Control Sample Levels

ELEMENT	LCS Level (ug/l)	Matrix Spike Level (ug/l)
Aluminum	2000	2000
Antimony	500	500
Arsenic	2000	2000
Barium	2000	2000
Beryllium	50	50
Cadmium	50	50
Calcium	50000	50000
Chromium	200	200
Cobalt	500	500
Copper	250	250
Iron	1000	1000
Lead	500	500
Lithium	1000	1000
Magnesium	50000	50000
Manganese	500	500
Molybdenum	1000	1000
Nickel	500	500
Phosphorous	10000	10000
Potassium	50000	50000
Selenium	2000	2000
Silver	50	50
Sodium	50000	50000
Strontium	1000	1000
Thallium	2000	2000
Vanadium	500	500
Zinc	500	500
Boron	1000	1000
Silicon	10000	10000
Tin	2000	2000
Titanium	1000	1000
Bismuth	1000	1000
Zirconium	1000	1000
Tellurium	1000	1000
Thorium	1000	1000
Uranium	1000	1000
Tungsten	1000	1000
Palladium	1000	1000

TABLE IV. ICP Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	100000	200	400	25000	50000
Antimony	10000	60	120	1000	5000
Arsenic	10000	300	600	1000	5000
Barium	10000	200	*20*	1000	5000
Beryllium	10000	5	10	1000	5000
Cadmium	10000	5	10	1000	5000
Calcium	100000	5000	10000	25000	50000
Chromium	10000	10	20	1000	5000
Cobalt	10000	50	100	1000	5000
Copper	10000	25	*20*	1000	5000
Iron	100000	100	*100*	25000	50000
Lead	10000	100	200	1000	5000
Lithium	10000	50	100	1000	5000
Magnesium	100000	5000	10000	25000	50000
Manganese	10000	15	*20*	1000	5000
Molybdenum	10000	40	80	1000	5000
Nickel	10000	40	80	1000	5000
Phosphorous	10000	300	600	1000	5000
Potassium	100000	5000	10000	25000	50000
Selenium	10000	250	500	1000	5000
Silver	2000	10	20	500	1000
Sodium	100000	5000	10000	25000	50000
Strontium	10000	50	100	1000	5000
Thallium	20000	2000	4000	5000	10000
Vanadium	10000	50	100	1000	5000
Zinc	10000	20	40	1000	5000
Boron	10000	200	400	1000	5000
Silicon	10000	500	1000	1000	5000
Tin	10000	100	200	1000	5000
Titanium	10000	50	100	1000	5000
Bismuth	10000	200	400	1000	5000
Zirconium	10000	100	200	1000	5000
Tellurium	10000	500	1000	1000	5000
Thorium	10000	500	1000	1000	5000
Uranium	10000	500	1000	1000	5000
Tungsten	10000	500	1000	1000	5000
Palladium	10000	100	200	1000	5000

TABLE IVA. Trace Calibration and Calibration Verification Standards

Element	Calibration Level	RL (ug/L)	CRI (ug/L)	ICV (ug/L)	CCV (ug/L)
Aluminum	50000	200	*100*	12500	25000
Antimony	1000	10	*10*	250	500
Arsenic	1000	10	*10*	250	500
Barium	4000	10	20	1000	2000
Beryllium	4000	5	10	1000	2000
Cadmium	1000	2	*2*	250	500
Calcium	100000	5000	10000	25000	50000
Chromium	4000	5	10	1000	2000
Cobalt	4000	50	*40*	1000	2000
Copper	4000	25	*20*	1000	2000
Iron	50000	100	*100*	12500	25000
Lead	1000	3	6	250	500
Magnesium	100000	5000	10000	25000	50000
Manganese	4000	15	*20*	1000	2000
Molybdenum	4000	40	*20*	1000	2000
Nickel	4000	40	80	1000	2000
Potassium	100000	5000	10000	25000	50000
Selenium	1000	5	10	250	500
Silver	2000	5	10	500	1000
Sodium	100000	5000	10000	25000	50000
Thallium	2000	10	20	500	1000
Vanadium	4000	50	*40*	1000	2000
Zinc	4000	20	40	1000	2000

TABLE V. Interference Check Sample Concentrations*

Element	ICSA (ug/L)	ICSAB (ug/L)
Aluminum	500000	500000
Antimony	-	1000
Arsenic	-	1000
Barium	-	500
Beryllium	-	500
Cadmium	-	1000
Calcium	500000	500000
Chromium	-	500
Cobalt	-	500
Copper	-	500
Iron	200000	200000
Lead	-	1000
Magnesium	500000	500000
Manganese	-	500
Molybdenum	-	1000
Nickel	-	1000
Potassium	-	10000
Selenium	-	1000
Silver	-	1000
Sodium	-	10000
Thallium	-	10000
Vanadium	-	500
Zinc	-	1000
Tin	-	1000

* Custom Quanterra solutions contain analytes common to all Quanterra facilities. Non-routine elements not listed above must be spiked into the ICSAB at 1000 ug/L.

TABLE VI. TCLP Reporting Limits, Regulatory Limits and Matrix Spike Levels

ELEMENT	RL (ug/L)	Regulatory Limit (ug/L)	Spike Level (ug/L)
Arsenic	500	5000	5000
Barium	10000	100000	50000
Cadmium	100	1000	1000
Chromium	500	5000	5000
Lead	500	5000	5000
Selenium	250	1000	1000
Silver	500	5000	1000

TABLE VII. Summary Of Quality Control Requirements

QC PARAMETER	FREQUENCY *	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Reanalysis of High Standard (HCAL)	Beginning of every analytical run, after CRI.	95 - 105 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICV	Beginning of every analytical run.	Method 200.7: 95 - 105 % recovery. Method 6010A: 90 - 110 % recovery.	Terminate analysis; Correct the problem; Recalibrate.
ICB	Beginning of every analytical run, immediately following the ICV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate.
CCV	Every 10 samples and at the end of the run.	90 - 110 % recovery	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCV.
CCB	Immediately following each CCV.	The result must be within +/- RL from zero.	Terminate analysis; Correct the problem; Recalibrate and rerun all samples not bracketed by acceptable CCB.
ICSA	Beginning and end of every run and every 8 hours.	Analyte results must be within +/- 2x RL from zero for analytes with RL ≤ 10 ug/L.	See Section 9.9.
ICSAB	Immediately following each ICSA.	Results must be within 80 - 120% recovery.	See Section 9.9.
CRI	Immediately following ICB.	Results must be within 50 - 150% recovery.	Terminate analysis; Correct the problem; Recalibrate.

* See Sections 11.11 and 11.12 for exact run sequence to be followed.

TABLE VII. Summary of Quality Control Requirements (Continued)

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Serial Dilution	One per prep batch.	For samples > 40x IDL, dilutions must agree within 10%.	Narrate the possibility of physical or chemical interference.
Method Blank	One per sample preparation batch of up to 20 samples.	The result must be less than or equal to the RL. Common lab contaminants may be accepted up to 2x the RL after consultation with the client (See 9.2). Sample results greater than 20x the blank concentration are acceptable. Samples for which the contaminant is < RL may not require redigestion or reanalysis (see Section 9.2).	Redigest and reanalyze samples. Note exceptions under criteria section. See Section 9.2 for additional requirements.
Laboratory Control Sample (LCS)	One per sample preparation batch of up to 20 samples.	Aqueous LCS must be within 80 - 120% recovery or in-house control limits. Samples for which the contaminant is < RL and the LCS results are > 120% may not require redigestion or reanalysis (see Section 9.3)	Terminate analysis; Correct the problem; Redigest and reanalyze all samples associated with the LCS.
Matrix Spike	One per sample preparation batch of up to 20 samples.	80 - 120 % recovery. If the MS/MSD is out for an analyte, it must be in control in the LCS. For TCLP See Section 11.17.	In the absence of client specific requirements, flag the data; no flag required if the sample level is > 4x the spike added. For TCLP see Section 11.17.

QC PARAMETER	FREQUENCY	ACCEPTANCE CRITERIA	CORRECTIVE ACTION
Matrix Spike Duplicate	See Matrix Spike	80 - 120 % recovery; RPD ≤ 20% (See MS).	See Corrective Action for Matrix Spike.

APPENDIX B

QUANTERRA ICP DATA REVIEW CHECKLIST

Quanterra ICP Data Review Checklist

Run/Project Information:

Run Date: _____ Analyst: _____ Instrument: _____
Prep Batches Run: _____

Circle Methods used: 6010A / 200.7: CORP-MT-0001 Rev 1
CLP : CORP-MT-0002 Rev 1

Review Items

A. Calibration/Instrument Run QC	Yes	No	N/A	2nd Level
1. Instrument calibrated per manufacturer's instructions and at SOP specified levels ?				
2. ICV/CCV analyzed at appropriate frequency and within control limits ? (*ICV:* 6010A, CLP = 90 - 110%, 200.7 = 95 -105%) *(CCV: 90 - 110%)*				
3. ICB/CCB analyzed at appropriate frequency and within +/- RL or +/- CRDL (CLP) ?				
4. High Std. (HCAL) reanalyzed before samples and recovered within QC limits ? (6010A/200.7 95-105%, CLP- N/A)				
5. CRI run and recovered within QC limits ? (+/- 50% for non-CLP)				
6. ICSA/ICSAB run at required frequency and within SOP limits ?				
B. Sample Results				
1. Were samples with concentrations > the linear range for any parameter diluted and reanalyzed ?				
2. All reported results bracketed by in control QC ?				
3. Sample analyses done within holding time ?				
C. Preparation/Matrix QC				
1. LCS done per prep batch and within QC limits ?				
2. Method blank done per prep batch and < RL or CRDL (CLP) ?				
3. MS run at required frequency and within limits ?				
4. MSD or DU run at required frequency and RPD within SOP limits ?				
5. Serial dilution done per prep batch (or per SDG for CLP) ?				
6. Post digest spike analyzed if required (CLP only) ?				
D. Other				
1. Are all nonconformances documented appropriately ?				
2. Current IDL/LR/IEC data on file ?				
3. Calculations checked for error ?				
4. Transcriptions checked for error ?				
5. All client/project specific requirements met ?				
6. Date/time of analysis verified as correct ?				

Analyst: _____ Date: _____
Comments: _____

2nd Level Reviewer : _____ Date: _____
Comments: _____

APPENDIX C

TJA CALIBRATION RECOMMENDATION

APPENDIX D

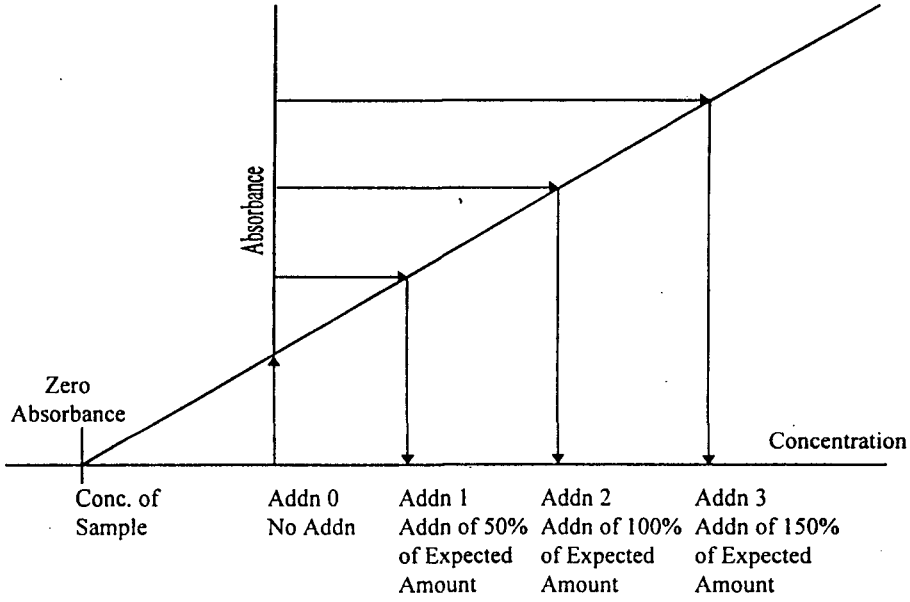
MSA GUIDANCE

Appendix D. MSA Guidance

Method of Standard Addition

Four equal volume aliquots of sample are measured and known amounts of standards are added to three aliquots. The fourth aliquot is the unknown and no standard is added to it. The concentration of standard added to the first aliquot should be 50% of the expected concentration. The concentration of standard added to the second aliquot should be 100% of the expected concentration and the concentration of standard added to the third aliquot should be 150% of the expected concentration. The volume of the unspiked and spiked standard should be the same.

In order to determine the concentration of analyte in the sample, the analytical value of each solution is determined and a plot or linear regression performed. On the vertical axis the analytical value is plotted versus the concentrations of the standards on the horizontal axis. An example plot is shown in Figure 1. When the resulting line is extrapolated back to zero absorbance, the point of interception of the horizontal axis is the concentration of the unknown.



For the method of standard additions to be correctly applied, the following limitations must be taken into consideration:

- The plot of the sample and standards must be linear over the concentration range of concern. For best results, the slope of the curve should be similar to that of a plot of the aqueous standard curve.
- The effect of the interference should not vary as the ratio of the standard added to the sample matrix changes.

APPENDIX E

TROUBLESHOOTING GUIDE

APPENDIX E. TROUBLESHOOTING GUIDE

Problem	Possible Cause/ Solution
High Blanks	Increase rinse time Clean or replace tip Clean or replace torch Clean or replace sample tubing Clean or replace nebulizer Clean or replace mixing chamber Lower Torch
Instrument Drift	RF not cooling properly Vacuum level is too low Replace torch (Crack) Clean or replace nebulizer (blockage) Check room temperature (changing) Replace pump tubing Room humidity too high Clean torch tip (salt buildup) Check for argon leaks Adjust sample carrier gas Reprofile Horizontal Mirror Replace PA tube
Erratic Readings, Flickering Torch or High RSD	Check for argon leaks Adjust sample carrier gas Replace tubing (clogged) Check drainage(back pressure changing) Increase uptake time (too short) Increase flush time (too short) Clean nebulizer, torch or spray chamber Increase sample volume introduced Check that autosampler tubes are full Sample or dilution of sample not mixed Increase integration time (too short) Realign torch Reduce amount of tubing connectors
Cu/Mn Ratio Outside Limits or Low Sensitivity	Plasma conditions changed Clean nebulizer, torch or spray chamber Replace tubing (clogged) Realign torch Check IEC's
Standards reading twice normal absorbance or concentration	Incorrect standard used Incorrect dilution performed

APPENDIX F

CONTAMINATION CONTROL GUIDELINES

APPENDIX F. CONTAMINATION CONTROL GUIDELINES

The following procedures are strongly recommended to prevent contamination:

All work areas used to prepare standards and spikes should be cleaned before and after each use.

All glassware should be washed with detergent and tap water and rinsed with 1:1 nitric acid followed by deionized water.

Proper laboratory housekeeping is essential in the reduction of contamination in the metals laboratory. All work areas must be kept scrupulously clean.

Powdered or Latex Gloves must not be used in the metals laboratory since the powder contains silica and zinc as well as other metallic analytes. Only vinyl or nitrile gloves should be used in the metals laboratory.

Glassware should be periodically checked for cracks and etches and discarded if found. Etched glassware can cause cross contamination of any metallic analytes.

Autosampler trays should be covered to reduce the possibility of contamination. Trace levels of elements being analyzed in the samples can be easily contaminated by dust particles in the laboratory.

The following are helpful hints in the identification of the source of contaminants:

Yellow pipet tips and volumetric caps can sometimes contain cadmium.

Some sample cups have been found to contain lead.

The markings on glass beakers have been found to contain lead. If acid baths are in use for glassware cleaning, they should be periodically checked for contaminants since contaminant concentrations will increase over time.

New glassware especially beakers can be a source of silica and boron.

Reagents or standards can contain contaminants or be contaminated with the improper use of a pipette.

Improper cleaning of glassware can cause contamination.

Latex gloves contain over 500 ppb of zinc.

APPENDIX G

PREVENTIVE MAINTENANCE

APPENDIX G. PREVENTIVE MAINTENANCE

A maintenance log is used to record when maintenance is performed on instruments. When an instrument problem occurs indicate the date, time and instrument number, then identify the problem and corrective action in the maintenance log.

The following procedures are required to ensure that that the instrument is fully operational.

Daily	Change sample pump tubing and pump windings Check argon gas supply level Check rinse solution and fill if needed Check waste containers and empty if needed Check sample capillary tubing is clean and in good condition Check droplet size to verify nebulizer is not clogged. Check sample flow for cross flow nebulizer Check Cu/Mn ratio-should be 30% of value at date that IECs were performed Check pressure for vacuum systems
As Needed	Clean plasma torch assembly to remove accumulated deposits Clean nebulizer and drain chamber; keep free-flowing to maintain optimum performance Replace peristaltic pump tubing, sample capillary tubing and autosampler sipper probe
Weekly	Apply silicon spray on autosampler tracks Check water level in coolflow
Monthly	Clean air filters on back of power unit to remove dust Check D mirror for air instruments
Bi-yearly	Change oil for vacuum systems Replace coolant water filter (may require more or less frequently depending on quality of cooling water)

APPENDIX B-9

TOTAL ORGANIC CARBON

QUANTERRA INCORPORATED

SOP CHANGE FORM

SOP NUMBER:NC-WC-0018

SOP TITLE:Total Organic Carbon (TOC) Analysis for Non-Waters

SOP SECTION(S) AFFECTED BY CHANGE:4.3

REASON FOR ADDITION OR CHANGE:Failed to remove this section in the recent previous revision dated 1/10/97

CHANGE EFFECTIVE FROM: (DATE):1/10/97

CHANGE OR ADDITION (SPECIFY SECTION; USE ADDITIONAL SHEETS IF NECESSARY)
[Identify change or issue document change with italics or change bar.]

Delete section 4.3: "Inorganic carbon interferes, but is eliminated by acidification and heating."

SUBMITTED BY/DATE:Opal Johnson 2/27/97

*APPROVED BY:

Technical Reviewer Signature Liz Belcon Date 2-28-97

Environmental Health & Safety Signature Date 2-28-97

QA Signature Date 2/28/97

Management Signature Date 2/28/97

**Must be same signature authorities of SOP being revised.*

n:\qaqc\qaform\change\ncwc18_2.doc

QUANTERRA® STANDARD OPERATING PROCEDURE

TITLE: TOTAL ORGANIC CARBON (TOC) ANALYSIS FOR NON-WATERS

(SUPERSEDES: REVISION 1, DATED 01/10/97)

Prepared by: _____

Reviewed by: _____
Technology Specialist

Approved by: _____ 2/28/97
Quality Assurance Manager

Approved by: _____ 2-28-97
Environmental Health and Safety Coordinator

Approved by: _____ 2-28-97
Laboratory Director

TABLE OF CONTENTS

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Total Organic Carbon in liquid, oils, sludge, soil, and sediment samples. It is based on Methods of Soil Analysis, Walkley-Black. The working linear range is 100 to 15,000 mg/kg.

1.2. This document accurately reflects current laboratory standard operating procedures (SOP) as of the date above. All facility SOPs are maintained and updated as necessary by the laboratory QA department.

2. SUMMARY OF METHOD

2.1. An aliquot of a solid sample is treated with excess potassium dichromate and concentrated sulfuric acid. After treatment, the solution is backtitrated with ferrous sulfate to determine the amount of dichromate reduced during digestion.

3. DEFINITIONS

3.1. Refer to the glossary in the Quality Assurance Management Plan (QAMP).

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. Specific selection of reagents may be required to avoid introduction of contaminants.

4.2. Chloride and iron give a positive interference. Chloride may be totally or partially eliminated by the addition of mercuric sulfate.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained from the Material Safety Data Sheets (MSDS) maintained in the laboratory. The following specific hazard is known:

5.3.1. The following material is known to be **corrosive: Sulfuric acid.**

5.4. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples must be opened, transferred and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.5. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operation will permit.

5.6. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

6. EQUIPMENT AND SUPPLIES

6.1. Buret: 25 mL Class A

6.2. Analytical balance: capable of weighing to ± 0.0001 g

6.3. Top loading balance: capable of weighing to ± 0.01 g

6.4. Amber bottles

6.5. Beakers: various

6.6. Graduated cylinders: various

6.7. Volumetric pipettes: various , Class A

6.8. Erlenmeyer flasks: various

6.9. Whatman #4 filter paper

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Sulfuric Acid ($H_2 SO_4$): concentrated, Tracepur grade

7.1.2. Ferroin indicator, purchased

7.1.3. Potassium Dichromate ($K_2Cr_2O_7$): primary standard grade

7.1.4. 1N Potassium Dichromate Solution: Accurately weigh 49.04 g of potassium dichromate (dried overnight at 105°C) in a liter volumetric flask and dilute to volume. Store in amber bottle and refrigerate. Replace after six months.

7.1.5. Ferrous Sulfate ($FeSO_4 \bullet 7 H_2O$): reagent grade

7.1.6. 0.5 N Ferrous Sulfate Titrant: Accurately weigh 140 g of $FeSO_4 \bullet 7H_2O$ into a 1 liter volumetric flask and dissolve with 500 mL reagent water. Carefully add 15 mL of concentrated sulfuric acid and allow to cool. Dilute to volume with reagent water. Store in amber bottle and refrigerate.

7.1.7. Mercuric Sulfate ($HgSO_4$): reagent grade

7.2. Standards

7.2.1. Laboratory Control Sample

7.2.1.1. Potassium Hydrogen Phthalate ($KHC_8H_4O_4$), purchased

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Samples are stored in a glass container at 4°C ± 2°C.

8.2. Samples are not chemically preserved. In lieu of no guidance, holding time is based on water requirements.

8.3. The holding time is twenty-eight days from sampling to analysis.

9. QUALITY CONTROL

9.1. Batch Definition

9.1.1. A batch is a group of no greater than 20 samples excluding QC samples (LCS, Method Blank, MS, MSD) which are processed similarly, with respect to the procedure. All sample setups must be initiated within a 24 hour period from the initial preparation or extraction and without interruption of the process. All samples within the batch must be treated with the same lots of reagents and the same processes.

9.2. Method Blank

9.2.1. One method blank (MB) must be processed with each preparation batch. The method blank consists of reagent water containing all reagents specific to the method that is carried through the entire analytical procedure, including preparation and analysis. The method blank is used to identify any system and process interferences or contamination of the analytical system that may lead to the reporting of elevated analyte concentrations or false positive data. The method blank should not contain any analyte of interest at or above the reporting limit.

9.2.2. A reagent water blank consisting of 200 mL reagent water is be prepared and analyzed with each analytical batch of samples.

9.2.3. Corrective Action for Blanks

9.2.3.1. If the analyte level in the method blank exceeds the reporting limit for the analytes of interest in the sample, all associated samples are reprepared and reanalyzed. If this is not possible due to limited sample quantity or other considerations, the corresponding sample data **must be addressed in the project narrative.**

9.2.3.2. If there is no analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. **Such action must be taken in consultation with the client and must be addressed in the project narrative.**

9.3. Laboratory Control Sample (LCS)

9.3.1. One LCS must be processed with each preparation batch. The LCS must be carried through the entire analytical procedure. The LCS is used to monitor the accuracy of the analytical process. On-going monitoring of the LCS results

provides evidence that the laboratory is performing the method within acceptable accuracy and precision guidelines.

9.3.2. A midrange LCS using 0.02 g of potassium hydrogen phthalate is prepared and analyzed with each batch of samples.

9.3.3. Corrective Action for LCS

9.3.3.1. If any analyte is outside established control limits the system is out of control and corrective action must occur.

9.3.3.2. Corrective action will be repreparation and reanalysis of the batch unless the client agrees that other corrective action is acceptable.

9.4. Duplicates

9.4.1. Sample duplicates are performed at a frequency of 10% or one per batch which ever is more frequent and must meet laboratory-specific limits for precision.

10. CALIBRATION AND STANDARDIZATION

10.1. The ferrous sulfate titrant is standardized daily as follows.

10.1.1. Pipet 10.0 mL of 1.00 N potassium dichromate solution into a 250 mL Erlenmeyer flask and add 90 mL reagent water.

10.1.2. Carefully add 30 mL of concentrated sulfuric acid and allow to cool completely.

10.1.3. Add 2-3 drops of ferroin indicator.

10.1.4. Titrate with 0.5 N ferrous sulfate titrant to a reddish-brown endpoint or to the first color change after reaching an emerald-green color.

10.1.5. Calculate the normality using the following equation.

$$N = \frac{10}{mL\ ferrous\ sulfate}$$

10.1.6. Repeat steps 10.1.1 through 10.1.5 two more times.

10.1.7. The average of the triplicate standardization is used.

11. PROCEDURE

11.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and is approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.3. Sample Preparation

11.3.1. Physical Preparation

11.3.1.1. Mix the sample thoroughly before selecting a portion for analysis.

11.3.1.2. Discard any foreign objects such as sticks, leaves, and rocks.

11.3.2. Analytical Preparation

11.3.2.1. Weigh an aliquot of soil of 2.50 g to the nearest 0.01 g (use less sample if TOC is known to be high). Record the weight on the analytical logsheet.

11.3.2.2. Place sample in a 500 mL Erlenmeyer flask and add 10.0 mL of 1 N potassium dichromate.

11.3.2.3. Under a hood, carefully add 20 mL of concentrated sulfuric acid and gently swirl for one minute.

11.3.2.4. Add 200 mL of reagent water and swirl to mix. If necessary, filter sample through Whatman #4 filter.

11.4. Sample Analysis Procedure

11.4.1. Add 2-3 drops ferroin indicator.

11.4.2. Titrate with 0.5 N ferrous Sulfate Solution to a reddish-brown endpoint or first color change after reaching an emerald-green color.

11.4.2.1. If the digestate of the sample is already green or reddish-brown after the addition of the ferroin indicator, the sample needs to be re-extracted with a smaller sample amount.

11.4.3. Document the amount of titrant on the analytical logsheet.

11.5. Analytical Documentation

11.5.1. Record all analytical information in the analytical logbook/logsheet, including the analytical data from standards, blanks, LCSs, MS/MSDs, and any corrective actions or modifications to the method.

11.5.2. All standards are logged into a department standard logbook. All standards are assigned an unique number for identification. Logbooks are reviewed by the supervisor or designee.

11.5.3. Documentation such as all associated instrument printouts (final runs, screens, reruns, QC samples, etc.) and daily calibration data corresponding to all final runs is available for each data file.

11.5.4. Sample results and associated QC are entered into the LIMs after final technical review.

12. DATA ANALYSIS AND CALCULATIONS

12.1. **Total Organic Carbon, mg/kg =**

$$\left[\frac{\textbf{[(10)(N Potassium Dichromate) - (mL Ferrous Sulfate) (N Ferrous Sulfate)]}}{\textbf{Weight of Soil (g)}} \textbf{ x 3000}\right] \times \textbf{ 1.3}$$

Where:

1.3 = Correction factor recommended in method

12.2. $$\textbf{TOC, \%} = \frac{\textbf{mg / kg}}{\textbf{10,000}}$$

12.3. $$\textbf{LCS, \%} = \frac{\textbf{TOC, \%}}{\textbf{61.152 (true)}} \textbf{ x 100}$$

13. METHOD PERFORMANCE

13.1. Each laboratory must have initial demonstration of performance data on file and corresponding method detection limit files.

13.2. Training Qualifications:

13.2.1. The group/team leader has the responsibility to ensure that this procedure is performed by an associate who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Acid waste must be collected in clearly labeled acid waste containers.

15.2. Solid materials (gloves, soiled paper products, etc.) are placed in the solid debris container. Do not put liquids in the solid waste container.

15.3. Refer to the Laboratory Sample and Waste Disposal plan.

15.4. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices of Quanterra. They must have training on the hazardous waste disposal practices upon initial assignment to these tasks, followed by an annual refresher training.

16. REFERENCES

16.1. References

16.1.1. Methods of Soil Analysis, 1982 Second Edition Method 29-3.5.2 Walkley-Black Procedure.

17. MISCELLANEOUS (TABLES, APPENDICES, ETC...)

17.1. Reporting limits

17.1.1. The lower reporting limit (RL) for undiluted samples is 100 mg/kg.

17.1.2. If samples require dilution or smaller volumes than specified in this method, the RL will be elevated.

17.2. Troubleshooting guide

17.2.1. When interferences as described in Section 4 are encountered or suspected, treat the sample as specified in that section.

17.2.2. If a high level of TOC is suspect (black sample), a smaller amount will be required.

APPENDIX B-10

DETERMINATION OF VOLATILE ORGANICS BY GC/MS

Controlled Copy UNCONTROLLED COPY
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SOP No. CORP-MS-0002
Revision No. 1
Revision Date: 01/31/96

QUANTERRA STANDARD OPERATING PROCEDURE

TITLE: DETERMINATION OF VOLATILE ORGANICS BY GC/MS BASED ON METHODS 8240B AND 8260A

(SUPERSEDES: REVISION 0)

Prepared by: Richard Burrows

Reviewed by: _____
Technology Standardization Committee

Approved by: _____
Director, Quality Assurance

Approved by: _____
Director, Environmental Health and Safety

Approved by: _____
Management

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TABLE OF CONTENTS

LIST OF TABLES

1. SCOPE AND APPLICATION

1.1. This method is applicable to the determination of Volatile Organic Compounds in waters, wastewaters, soils, sludges and other solid matrices. Standard analytes are listed in Tables 5 and 6.

1.2. This SOP is applicable to methods 8240B (capillary column) and 8260A.

1.3. This method can be used to quantify most volatile organic compounds that have boiling points below 200°C and are insoluble or slightly soluble in water. Volatile water soluble compounds can be included in this analytical technique; however, for more soluble compounds, quantitation limits are approximately ten times higher because of poor purging efficiency.

1.4. The method is based upon a purge and trap, gas chromatograph/mass spectrometric (GC/MS) procedure. Directions are provided for analysis based on methods 8240B and 8260A. The approximate working range is 5 to 200 µg/L for 5 mL waters, 1 to 60 µg/L for 25 mL purge waters, 5 to 200 µg/kg for low-level soils, and 630 to 25,000 µg/kg for medium-level soils. Reporting limits are listed in Tables 1, 3 and 14.

1.5. Method performance is monitored through the use of surrogate compounds, matrix spike/matrix spike duplicates, and laboratory control spike samples.

2. SUMMARY OF METHOD

2.1. The differences between method 8240B and 8260A as performed at Quanterra are summarized here.

2.1.1. Method 8240 was written as a packed column method; however, Quanterra has modified the method to use capillary columns for improved performance. Accordingly, when the surrogates and internal standards listed in this SOP are used, methods 8240B and 8260A are similar except for the minimum response factors listed in Table 11. ***Both*** methods may use 5 or 25 mL purge volumes, depending on the detection limits required, and both methods have the same calibration criteria.

2.1.2. When a method 8260A analysis is requested, a target compound list based on that contained in method 524.2 is frequently requested. One of the target compounds, bromochloromethane, is a method 8240B internal standard, so alternative surrogates and internal standards must be used. Quanterra's standard analyte list, surrogates and internal standards for method 8260A (drinking water analyte list) are listed in Appendix A, Tables 14, 15, and 16.

2.2. The volatile compounds are introduced into the gas chromatograph by the purge and trap method. The components are separated via the chromatograph and detected using a mass spectrometer which is used to provide both qualitative and quantitative information.

2.3. If the above sample introduction techniques are not applicable, a portion of the sample is dispersed in methanol to dissolve the volatile organic constituents. A portion of the methanolic solution is combined with water in a purging chamber. It is then analyzed by purge and trap.

2.4. In the purge and trap process, an inert gas is bubbled through the solution at ambient temperature (40°C for soils) and the volatile components are efficiently transferred from the aqueous phase to the vapor phase. The vapor is swept through a sorbant column where the volatile components are trapped. After purging is completed, the sorbant column (trap) is heated and backflushed with inert gas to desorb the components onto a gas chromatographic column. The gas chromatographic column is then heated to elute the components which are detected with a mass spectrometer.

2.5. Qualitative identifications are confirmed by analyzing standards under the same conditions used for samples and comparing the resultant mass spectra and GC retention times. Each identified component is quantified by relating the MS response for an appropriate selected ion produced by that compound to the MS response for another ion produced by an internal standard.

3. DEFINITIONS

3.1. Batch

The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. Using this method, each BFB analysis will normally start a new batch. If no changes to instrumental parameters are made, a batch may extend for a maximum of 24 hours. Batches for medium level soils are defined at the sample preparation stage and may be analyzed on multiple instruments over multiple days, although reasonable effort should be made to keep the samples together.

3.1.1. The Quality Control batch must contain a matrix spike/spike duplicate (MS/MSD), a Laboratory Control Sample (LCS), and a method blank. In some cases, at client request, the MS/MSD may be replaced with a matrix spike and sample duplicate. Refer to the Quanterra QC Program document (QA-003) for further details of the batch definition.

3.2. Method Blank

A method blank consisting of all reagents added to the samples must be analyzed with each batch of samples. The method blank is used to identify any background interference or contamination of the analytical system which may lead to the reporting of elevated concentration levels or false positive data.

3.3. Laboratory Control Sample (LCS)

Laboratory Control Samples are well characterized, laboratory generated samples used to monitor the laboratory's day-to-day performance of routine analytical methods. The LCS, spiked with a group of target compounds representative of the method analytes, is used to monitor the accuracy of the analytical process, independent of matrix effects. Ongoing monitoring of the LCS results provides evidence that the laboratory is performing the method within accepted QC guidelines for accuracy and precision.

3.4. Surrogates

Surrogates are organic compounds which are similar to the target analyte(s) in chemical composition and behavior in the analytical process, but which are not normally found in environmental samples. Each sample, blank, LCS, and MS/MSD is spiked with surrogate standards. Surrogate spike recoveries must be evaluated by determining whether the concentration (measured as percent recovery) falls within the required recovery limits.

3.5. Matrix Spike/Matrix Spike Duplicate (MS/MSD)

A matrix spike is an environmental sample to which known concentrations of target analytes have been added. A matrix spike duplicate is a second aliquot of the same sample which is prepared and analyzed along with the sample and matrix spike. Matrix spikes and duplicates are used to evaluate accuracy and precision in the actual sample matrix.

3.6. Calibration Check Compound (CCC)

CCCs are a representative group of compounds which are used to evaluate initial calibrations and continuing calibrations. Relative percent difference for the initial calibration and % drift for the continuing calibration response factors are calculated and compared to the specified method criteria.

3.7. System Performance Check Compounds (SPCC)

SPCCs are compounds which are sensitive to system performance problems and are used to evaluate system performance and sensitivity. A response factor from the continuing calibration is calculated for the SPCC compounds and compared to the specified method criteria.

4. INTERFERENCES

4.1. Method interferences may be caused by contaminants in solvents, reagents, glassware, and other processing apparatus that lead to discrete artifacts. All of these materials must be routinely demonstrated to be free from interferences under conditions of the analysis by running laboratory method blanks as described in the Quality Control section. The use of ultra high purity gases, pre-purged purified reagent water, and approved lots of purge and trap grade methanol will greatly reduce introduction of contaminants. In extreme cases the purging vessels may be pre-purged to isolate the instrument from laboratory air contaminated by solvents used in other parts of the laboratory.

4.2. Samples can be contaminated by diffusion of volatile organics (particularly methylene chloride and fluorocarbons) into the sample through the septum seal during shipment and storage. A field blank prepared from reagent water and carried through the sampling and handling protocol can serve as a check on such contamination.

4.3. Matrix interferences may be caused by non-target contaminants that are coextracted from the sample. The extent of matrix interferences will vary considerably from source to source depending upon the nature and diversity of the site being sampled.

4.4. Cross-contamination can occur whenever high-level and low-level samples are analyzed sequentially or in the same purge position on an autosampler. Whenever an unusually concentrated sample is analyzed, it should be followed by one or more blanks to check for cross-contamination. The purge and trap system may require extensive bake-out and cleaning after a high-level sample.

4.5. Some samples may foam when purged due to surfactants present in the sample. When this kind of sample is encountered an antifoaming agent (e.g., J.T. Baker's Antifoam B silicone emulsion) can be used. A blank spiked with this agent must be analyzed with the sample because of the non-target interferences associated with the agent.

5. SAFETY

5.1. Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2. The Chemical Hygiene Plan (CHP) gives details about the specific health and safety practices which are to be followed in the laboratory area. Personnel must receive training in the CHP, including the written Hazard Communication plan, prior to working in the laboratory. Consult the CHP, the Quanterra Health and Safety Policies and Procedures Manual, and available Material Safety Data Sheets (MSDS) prior to using the chemicals in the method.

5.3. Consult the Quanterra Health and Safety Policies and Procedures Manual for information on Personal Protective Equipment. Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan) and a laboratory coat must be worn in the lab. Appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately. Disposable gloves shall not be reused.

5.4. The health and safety hazards of many of the chemicals used in this procedure have not been fully defined, therefore each chemical compound should be treated as a potential health hazard. Additional health and safety information can be obtained from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.4.1. Chemicals that have been classified as carcinogens, or potential carcinogens, under OSHA include: Acrylonitrile, benzene, carbon tetrachloride, chloroform, 1,2-dibromo-3-chloropropane, 1,4-dichlorobenzene, and vinyl chloride.

5.4.2. Chemicals known to be flammable are: Methanol.

5.5. Exposure to chemicals must be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples should be opened, transferred, and prepared in a fume hood, or under other means of mechanical ventilation. Solvent and waste containers will be kept closed unless transfers are being made.

5.6. The preparation of standards and reagents will be conducted in a fume hood with the sash closed as far as the operations will permit.

5.7. All work must be stopped in the event of a known or potential compromise to the health and safety of a Quanterra associate. The situation must be reported **immediately** to a laboratory supervisor.

5.8. Laboratory personnel assigned to perform hazardous waste disposal procedures must have a working knowledge of the established procedures and practices outlined in the Quanterra Health and Safety Manual. These employees must have training on the hazardous waste disposal practices initially upon assignment of these tasks, followed by an annual refresher training.

6. EQUIPMENT AND SUPPLIES

6.1. Microsyringes: 10 µL and larger, 0.006 inch ID needle.

6.2. Syringe: 5 mL glass with luerlok tip, if applicable to the purging device.

6.3. Balance: Analytical, capable of accurately weighing 0.0001 g, and a top-loading balance capable of weighing 0.1 g

6.4. Glassware:

6.4.1. Vials: 20 mL with screw caps and Teflon liners.

6.4.2. Volumetric flasks: 10 mL and 100 mL, class A with ground-glass stoppers.

6.5. Spatula: Stainless steel.

6.6. Disposable pipets: Pasteur.

6.7. pH paper: Wide range.

6.8. Gases:

6.8.1. Helium: Ultra high purity, gr. 5, 99.999%.

6.8.2. Compressed air: Used for instrument pneumatics.

6.8.3. Liquid nitrogen: Used for cryogenic cooling if necessary.

6.9. Purge and Trap Device: The purge and trap device consists of the sample purger, the trap, and the desorber.

6.9.1. Sample Purger: The recommended purging chamber is designed to accept 5 mL samples with a water column at least 3 cm deep. The purge gas must pass through the water column as finely divided bubbles, each with a diameter of less than 3 mm at the origin. The purge gas must be introduced no more than 5 mm from the base of the water column. Alternative sample purge devices may be used provided equivalent performance is demonstrated.

6.9.2. Trap: The trap should be at least 25 cm long and have an inside diameter of at least 0.105 inch. Starting from the inlet, the trap should contain the following amounts of absorbents: 1/3 of 2,6-diphenylene oxide polymer (Tenax-GC, 60/80 mesh or equivalent), 1/3 of silica gel (Davison Chemical, 35/60 mesh, grade 15, or equivalent), and 1/3 coconut charcoal. It is recommended to use a trap that also 1.0 cm methyl silicone packing at the inlet to extend its life. If it is not necessary to analyze for any fluorocarbons then the charcoal phase can be replaced with the polymer. Other traps. such as Supelco's Vocarb 3000 and 4000, may be used if the Quality Control criteria are met.

6.9.3. Desorber: The desorber should be capable of rapidly heating the trap to 180°C. Many such devices are commercially available.

6.9.4. Sample Heater: A heater or heated bath capable of maintaining the purge device at 40°C is used for low level soil analysis only.

6.10. Gas Chromatograph/Mass Spectrometer System:

6.10.1. Gas Chromatograph: The gas chromatograph (GC) system must be capable of temperature programming. The system must include or be interfaced to a purge and trap device. All GC carrier gas lines must be made from stainless steel or copper tubing.

6.10.2. Gas Chromatographic Columns: Capillary columns are used. Some typical columns are listed below:

6.10.2.1. Column 1: 105m x 0.53 ID Rtx-624 with 3 µm film thickness.

6.10.2.2. Column 2: 75 m x 0.53 ID DB-624 widebore with 3 µm film thickness.

6.10.3. Mass Spectrometer: The mass spectrometer must be capable of scanning 35-300 AMU every two seconds or less, using 70 volts electron energy in the electron impact mode and capable of producing a mass spectrum that meets the required criteria when 50 ng of 4-Bromofluorobenzene (BFB) are injected onto the gas chromatograph column inlet.

6.10.4. GC/MS interface: In general glass jet separators are used but any interface that achieves all acceptance criteria may be used.

6.10.5. Data System: A computer system that allows the continuous acquisition and storage on machine readable media of all mass spectra obtained throughout the duration of the chromatographic program. The computer must have software that allows searching any GC/MS data file for ions of a specified mass and plotting such ion abundances versus time or scan number. This type of plot is defined as an Extracted Ion Current Profile (EICP). Software must also be available that allows integrating the abundances in any EICP between the specified time or scan-number limits. Also, for the non-target compounds, software must be available that allows for the comparison of sample spectra against reference library spectra. The most recent release of the NIST/EPA mass spectral library should be used as the reference library. The computer system must also be capable of backing up data for long-term off-line storage.

6.10.6. Cryogenic Cooling: Some columns require the use of liquid nitrogen to achieve the subambient temperature required for the proper separation of the gases.

7. REAGENTS AND STANDARDS

7.1. Reagents

7.1.1. Methanol: Purge and Trap Grade, High Purity

7.1.2. Reagent Water: High purity water that meets the requirements for a method blank when analyzed. (See section 9.4) Reagent water may be purchased as commercial distilled water and prepared by purging with an inert gas overnight. Other methods of preparing reagent water are acceptable.

7.2. Standards

7.2.1. Calibration Standard

7.2.1.1. Stock Solutions: Stock solutions may be purchased as certified solutions from commercial sources or prepared from pure standard materials as appropriate. These standards are prepared in methanol and stored in Teflon-sealed screw-cap bottles with minimal headspace at -10° to -20°C. Stock standards for gases must be replaced at least every week. Other stock standards must be replaced at least every 6 months.

7.2.1.2. Working standards: A working solution containing the compounds of interest prepared from the stock solution(s) in methanol. These standards are stored with minimal headspace and monitored for signs of degradation or evaporation, especially just prior to preparing calibration standards from them. The standards are stored for a maximum of one week.

Note: By definition in this SOP, a stock standard is one that is stored in the freezer and is only opened in order to prepare working standards. A working standard is opened more frequently in order to prepare the calibration, spiking or tuning standards used at the instrument. Stock and working standards may be at the same concentration.

7.2.1.3. Aqueous Calibration Standards are prepared in reagent water using the secondary dilution standards. These aqueous standards must be prepared daily.

7.2.1.4. If stock or secondary dilution standards are purchased in sealed ampoules they may be used up to the manufacturers expiration date. Once the ampoule is opened the expiration dates in Section 7.2.1.1 become effective.

7.2.2. Internal Standards: Internal standards are added to all samples, standards, and blank analyses. Refer to Table 7 for internal standard components.

7.2.3. Surrogate Standards: Refer to Table 8 for surrogate standard components and spiking levels.

7.2.4. Laboratory Control Sample Spiking Solutions: Refer to Table 9 for LCS components and spiking levels.

7.2.5. Matrix Spiking Solutions: The matrix spike contains the same components as the LCS. Refer to Table 9.

7.2.6. Tuning Standard: A standard is made up that will deliver 50 ng on column upon injection. A recommended concentration of 25 ng/µL of 4-Bromofluorobenzene in methanol is prepared as described in Sections 7.2.1.1 and 7.2.1.2.

8. SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1. Standard 40 mL glass screw-cap VOA vials with Teflon-faced silicone septa may be used for both liquid and solid matrices. Solid samples may also be collected in wide-mouth glass jars with Teflon-lined caps. Samples should be introduced into the containers with minimum agitation to avoid loss of volatile compounds. For liquid samples, each VOA vial should be filled without introduction of bubbles. Fill until there is a meniscus over the lip of the vial. The lid with septum (Teflon side toward the sample) should be tightened onto the vial. After tightening the lid, the vial should be inverted and tapped to check for air bubbles. If there are any air bubbles present the sample must be retaken. Sample containers for solid samples should be filled as completely as possible with minimum air space.

8.2. Water samples are preserved with HCl. Aromatic compounds are particularly susceptible to biodegradation at normal pH. The pH of the sample should be adjusted to less than 2 with HCl in the field at the time of sampling.

8.2.1. If the sample is unpreserved every reasonable effort should be made to analyze the sample within 7 days from sampling. The condition should be documented as an anomaly and the normal holding time applied. If the sample requires analysis of aromatic compounds the potential impact on the data must be documented.

8.3. All samples must be iced or refrigerated at 4° ± 2°C from the time of collection until analysis or extraction.

8.4. For shipping information, see the facility Sample Procurement Protocol SOP.

8.5. The holding time is fourteen days from sampling to the completion of analysis.

9. QUALITY CONTROL

9.1. Initial Demonstration of Capability

9.1.1. For the standard analyte list, the initial demonstration and method detection limit (MDL) studies described in Section 13 must be acceptable before analysis of samples may begin.

9.1.2. For non-standard analytes, a MDL study must be performed and calibration curve generated before analyzing any samples, unless lesser requirements are previously agreed to with the client. In any event, the minimum initial demonstration required is analysis of an extracted standard at the reporting limit and a single point calibration.

9.2. Control Limits

In-house historical control limits must be determined for surrogates, matrix spikes, and laboratory control samples (LCS). These limits must be determined at least annually. The recovery limits are mean recovery +/- 3 standard deviations for surrogates and LCS, and mean recovery +/- 2 standard deviations for matrix spikes. Precision limits for matrix spikes / matrix spike duplicates are 0 to mean relative percent difference + 2 standard deviations.

9.2.1. For medium level soils only, these limits do not apply to dilutions. Surrogate and matrix spike recoveries for medium level soils will be reported unless the dilution is more than 5X.

9.2.2. All surrogate, LCS, and MS recoveries (except for dilutions) must be entered into QuantIMS (when available) or other database so that accurate historical control limits can be generated. For tests without a separate extraction, surrogates and matrix spikes will be reported for all dilutions.

9.2.3. Refer to the QC Program document (QA-003) for further details of control limits.

9.3. Surrogates

Every sample, blank, and QC sample is spiked with surrogates. Surrogate recoveries in samples, blanks, and QC samples must be assessed to ensure that recoveries are within established limits. The compounds included in the surrogate spiking solutions are listed in Tables 8 and 16. If any surrogates are outside limits, the following corrective actions must take place (except for dilutions):

- Check all calculations for error.

- Ensure that instrument performance is acceptable.
- Recalculate the data and/or reanalyze if either of the above checks reveal a problem.
- Reprepare and reanalyze the sample or flag the data as "Estimated Concentration" if neither of the above resolves the problem.

The decision to reanalyze or flag the data should be made in consultation with the client. It is only necessary to reprepare/reanalyze a sample once to demonstrate that poor surrogate recovery is due to matrix effect, unless the analyst believes that the repeated out of control results are not due to matrix effect.

9.3.1. If the surrogates are out of control for the sample, matrix spike, and matrix spike duplicate, then matrix effect has been demonstrated for that sample and repreparation is not necessary. If the sample is out of control and the MS and/or MSD is in control, then reanalysis or flagging of the data is required.

9.3.2. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.4. Method Blanks

For each batch of samples, analyze a method blank. The method blank is normally analyzed immediately after the calibration standards. For low-level volatiles, the method blank consists of reagent water. For medium-level volatiles, the method blank consists of 9.0 mL of methanol. Surrogates are added and the method blank is carried through the entire analytical procedure. The method blank must not contain any analyte of interest at or above the reporting limit (except common laboratory contaminants, see below) or at or above 5% of the measured concentration of that analyte in the associated samples, whichever is higher.

- If the analyte is a common laboratory contaminant (methylene chloride, acetone, 2-butanone) the data may be reported with qualifiers if the concentration of the analyte is less than five times the reporting limit. Such action must be taken in consultation with the client.
- Reanalysis of samples associated with an unacceptable method blank is required when reportable concentrations are determined in the samples.
- If there is no target analyte greater than the RL in the samples associated with an unacceptable method blank, the data may be reported with qualifiers. Such action should be done in consultation with the client.

9.4.1. The method blank must have acceptable surrogate recoveries. If surrogate recoveries are not acceptable, the data must be evaluated to determine if the method blank has served the purpose of demonstrating that the analysis is free of contamination. If surrogate recoveries are low and there are reportable analytes in the associated samples re-extraction of the blank and affected samples will normally be required. Consultation with the client should take place.

9.4.2. If reanalysis of the batch is not possible due to limited sample volume or other constraints, the method blank is reported, all associated samples are flagged with a "B," and appropriate comments may be made in a narrative to provide further documentation.

9.4.3. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective actions.

9.5. Laboratory Control Samples (LCS)

For each batch of samples, analyze a LCS. The LCS is normally analyzed immediately after the method blank. The LCS contains a representative subset of the analytes of interest (See Table 9), and must contain the same analytes as the matrix spike. If any analyte or surrogate is outside established control limits, the system is out of control and corrective action must occur. Corrective action will normally be repreparation and reanalysis of the batch.

- If the batch is not re-extracted and reanalyzed, the reasons for accepting the batch must be clearly presented in the project records and the report. (An example of acceptable reasons for not reanalyzing might be that the matrix spike and matrix spike duplicate are acceptable, and sample surrogate recoveries are good, demonstrating that the problem was confined to the LCS.)

- If re-extraction and reanalysis of the batch is not possible due to limited sample volume or other constraints, the LCS is reported, all associated samples are flagged, and appropriate comments are made in a narrative to provide further documentation.

9.5.1. Refer to the Quanterra QC Program document (QA-003) for further details of the corrective action.

9.5.2. If full analyte spike lists are used at client request, it will be necessary to allow a percentage of the components to be outside control limits as this would be expected statistically. These requirements should be negotiated with the client.

9.6. Matrix Spikes

For each QC batch, analyze a matrix spike and matrix spike duplicate. Spiking compounds and levels are given in Table 9. Compare the percent recovery and relative percent difference (RPD) to that in the laboratory specific historically generated limits.

- If any individual recovery or RPD falls outside the acceptable range, corrective action must occur. The initial corrective action will be to check the recovery of that analyte in the Laboratory Control Sample (LCS). Generally, if the recovery of the analyte in the LCS is within limits, then the laboratory operation is in control and analysis may proceed. The reasons for accepting the batch must be documented.

- If the recovery for any component is outside QC limits for both the matrix spike/ spike duplicate and the LCS, the laboratory is out of control and corrective action must be taken. Corrective action will normally include reanalysis of the batch.

- If a MS/MSD is not possible due to limited sample, then a LCS duplicate should be analyzed. RPD of the LCS and LCSD are compared to the matrix spike limits.

- The matrix spike/duplicate must be analyzed at the same dilution as the unspiked sample, even if the matrix spike compounds will be diluted out.

9.7. Nonconformance and Corrective Action

Any deviations from QC procedures must be documented as a nonconformance, with applicable cause and corrective action approved by the facility QA Manager.

9.8. Quality Assurance Summaries

Certain clients may require specific project or program QC which may supersede these method requirements. Quality Assurance Summaries should be developed to address these requirements.

9.9. Quanterra QC Program

Further details of QC and corrective action guidelines are presented in the Quanterra QC Program document (QA-003). Refer to this document if in doubt regarding corrective actions.

10. CALIBRATION AND STANDARDIZATION

10.1. Summary

10.1.1. Prior to the analysis of samples and blanks, each GC/MS system must be tuned and calibrated. Hardware tuning is checked through the analysis of the 4-Bromofluoro-benzene (BFB) to establish that a given GC/MS system meets the standard mass spectral abundance criteria. The GC/MS system must be calibrated initially at a minimum of five concentrations (analyzed under the same BFB tune), to determine the linearity of the response utilizing target calibration standards. Once the system has been calibrated, the calibration must be verified each twelve hour time period for each GC/MS system. The use of separate calibrations is required for water and low soil matrices.

10.2. Recommended Instrument Conditions

10.2.1. General

Electron Energy:	70 volts (nominal)
Mass Range:	35–300 AMU
Scan Time:	to give at least 5 scans/peak, but not to exceed 1 second/scan
Injector Temperature:	200–250°C
Source Temperature:	According to manufacturer's specifications
Transfer Line	Temperature: 250–300°C
Purge Flow:	40 mL/minute
Carrier Gas	Flow: 15 mL/minute
Make-up Gas Flow:	25–30 mL/minute

10.2.2. Gas chromatograph suggested temperature program

10.2.2.1. BFB Analysis

Isothermal:	170°C

10.2.2.2. Sample Analysis

Initial Temperature:	40°C
Initial Hold Time:	4 minutes
Temperature Program:	8°C/minute
Final Temperature:	184°C
Second Temperature	Program: 40°C/minute
Final Temperature:	240°C

Final Hold Time: 2.6 minutes

10.3. Instrument Tuning

10.3.1. Each GC/MS system must be hardware-tuned to meet the abundance criteria listed in Table 10 for a maximum of a 50 ng injection or purging of BFB. Analysis must not begin until these criteria are met. These criteria must be met for each twelve-hour time period. The twelve-hour time period begins at the moment of injection of BFB.

10.4. Initial Calibration

10.4.1. A series of five initial calibration standards is prepared and analyzed for the target compounds and each surrogate compound. The calibration levels for a 5 mL purge are: 10, 20, 50, 100, and 200 µg/L. Certain analytes are prepared at higher concentrations due to poor purge performance. Calibration levels for a 25 mL purge are 2, 5, 10, 30, and 60 µg/L. Again, some analytes are prepared at higher levels. Tables 2, 4, and 17 list the calibration levels for each analyte.

10.4.2. It may be necessary to analyze more than one set of calibration standards to encompass all of the analytes required for same tests. For example, the Appendix IX list requires the Primary standard (Table 5) and the Appendix IX standard (Table 6).

10.4.3. Internal standard calibration is used. The internal standards are listed in Tables 7 and 15. Target compounds should reference the nearest internal standard. In particular, the SPCC compounds bromoform and 1,1,2,2-tetrachloroethane reference chlorobenzene-d5, not 1,4-difluorobenzene, which was appropriate for packed column analysis. Note different internal standards are used for the method 8260A drinking water analyte list (Appendix A). Each calibration standard is analyzed and the response factor (RF) for each compound is calculated using the area response of the characteristic ions against the concentration for each compound and internal standard. See equation 1, Section 12, for calculation of response factor.

10.4.4. The % RSD of the calibration check compounds (CCC) must be less than 30%. Refer to Table 12 for the CCCs.

10.4.4.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 30% criterion.

10.4.5. The average RF must be calculated for each compound. A system performance check is made prior to using the calibration curve. The five system performance check compounds (SPCC) are checked for a minimum average response factor. Refer to Table 11 for the SPCC compounds and required minimum response factors.

10.4.6. If the %RSD of ≥ 80% of the analytes in the calibration is ≤ 15%, then all analytes may use average response factor for calibration.

10.4.6.1. If the software in use is capable of routinely reporting curve coefficients for data valivation purposes, and the necessary calibration reports can be generated, then the analyst should evaluate analytes with %RSD > 15% for calibration on a curve. If if appears that substantially better accuracy would be obtained using quantitation from a curve (e.g. R^2 >0.995) then the appropriate curve should be used for quantitation.

10.4.6.2. If less than 80% of the analytes in the calibration have %RSD ≤ 15%, then calibration on a curve must be used for all analytes with %RSD > 15%. The analyst should consider instrument maintenance to improve the linearity of response.

10.4.7. Weighting of data points

In a linear or quadratic calibration fit, the points at the lower end of the calibration curve have less weight in determining the curve generated than points at the high concentration end of the curve. However, in environmental analysis, accuracy at the low end of the curve is very important. For this reason it is preferable to increase the weighting of the lower concentration points. $1/Concentration^2$ weighting (often called $1/X^2$ weighting) will improve accuracy at the low end of the curve and should be used if the data system has this capability.

10.4.8. If time remains in the 12-hour period initiated by the BFB injection before the initial calibration, samples may be analyzed. Otherwise, proceed to continuing calibration.

10.4.9. A separate five point calibration must be prepared for analysis of low level soils. Each standard is prepared as in Section 10.4.1, <u>except</u> that the standards are heated to 40°C for purging. All low-level soil samples, standards, and blanks must also be heated to 40°C for purging. Medium soil extracts should be analyzed using the water (unheated) calibration curve.

10.5. Continuing Calibration: The initial calibration must be verified every twelve hours.

10.5.1. Continuing calibration begins with analysis of BFB as described in Section 10.3. If the system tune is acceptable, the continuing calibration standard(s) are analyzed. The level 3 calibration standard is used as the continuing calibration.

10.5.2. The RF data from the standards are compared with the average RF from the initial five-point calibration to determine the percent drift of the CCC compounds. The calculation is given in equation 4, Section 12.3.4.

10.5.3. The % drift of the CCCs must be < 20% for the continuing calibration to be valid. The SPCCs are also monitored. The SPCCs must meet the criteria described in Table 11. In addition, the % drift of all anlalytes must be ≤ 50% with allowance for up to six target analytes to have % drift > 50%.

10.5.3.1. If none of the CCCs are required analytes, project specific calibration specifications must be agreed with the client. Otherwise, all CCCs must meet the 20% criterion.

10.5.3.2. Cyclohexanone, one of the components of the Appendix IX standard, is unstable in the calibration solution, forming 1,1-dimethoxycyclohexane. No calibration criteria are applied to cyclohexanone and quantitation is tentative. Cyclohexanone is included on the Universal Treatment Standard and FO-39 regulatory lists (but not on Appendix IX).

10.5.4. If the CCCs and or the SPCCs do not meet the criteria in Sections 10.5.3 and 10.5.4, the system must be evaluated and corrective action must be taken. The BFB tune and continuing calibration must be acceptable before analysis begins. Extensive corrective action such as a different type of column will require a new initial calibration.

10.5.5. Once the above criteria have been met, sample analysis may begin. **Initial calibration average RFs (or the calibration curve) will be used for sample quantitation, not the continuing calibration RFs.** Analysis may proceed until 12 hours from the injection of the BFB have passed. (A sample *desorbed* less than or equal to 12 hours after the BFB is acceptable.)

11. PROCEDURE

11.1. Procedural Variations

11.1.1. One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation shall be completely documented using a Nonconformance Memo and approved by a Supervisor or group leader and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.1.2. Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and corrective action described.

11.2. Preliminary Evaluation

11.2.1. Where possible, samples are screened by headspace or GC/MS off-tune analysis to determine the correct aliquot for analysis. Alternatively, an appropriate aliquot can be determined from sample histories.

11.2.2. Dilutions should be done just prior to the GC/MS analysis of the sample. Dilutions are made in volumetric flasks or in a 5 or 25 mL Luerlok syringe. Calculate the volume of reagent water required for the dilution. Fill the 5/25 mL syringe with reagent water, compress the water to vent any residual air and adjust the water volume to the desired amount. Adjust the plunger to the 5/25 mL mark and inject the proper aliquot of sample into the syringe. If the dilution required would use less than 5 µL of sample then serial dilutions must be made in volumetric flasks.

11.2.2.1. The diluted concentration is to be estimated to be in the upper half of the calibration range.

11.3. Sample Analysis Procedure

11.3.1. All analysis conditions for samples must be the same as for the calibration standards (including purge time and flow, desorb time and temperature, column temperatures, multiplier setting etc.).

11.3.2. Sample Preparation Procedure

11.3.2.1. Samples fall into three general categories: waters, low-level soils, and medium-level soils or wastes. For waters and low-level soils, no sample preparation is necessary. For those soils which contain greater than 1 mg/kg of individual purgeable compounds, a medium-level preparation is necessary.

11.3.3. All samples must be analyzed as part of a batch. The batch is a set of up to 20 samples of the same matrix processed using the same procedures and reagents within the same time period. The batch also must contain a MS/MSD, a LCS, and a method blank.

11.3.3.1. If there is insufficient time in the 12-hour tune period to analyze 20 samples, the batch may be continued into the next tune period. However, if any re-tuning of the instrument is necessary, or if a period of greater than 24 hours from the preceding BFB tune has passed, a new batch must be started. For medium level soils the batch is defined at the sample preparation stage.

11.3.3.2. One MS/MSD pair does not count towards the maximum 20 samples in the batch. Additional client requested MS/MSD samples do count towards the maximum 20 samples.

11.3.3.3. It is not necessary to reanalyze batch QC with reanalyses of samples. However, any reruns must be as part of a valid batch.

11.3.4. Water Samples

11.3.4.1. All samples and standard solutions must be at ambient temperature before analysis.

11.3.4.2. Fill a 5 or 25 mL syringe with the sample. If a dilution is necessary it may be made in the syringe if the sample aliquot is $\geq$ 5 µL. Check and document the pH of the remaining sample.

11.3.4.3. Add 250 ng of each internal and surrogate standard (10 µL of a 25 µg/mL solution, refer to Tables 7, 8, 15 and 16). The internal standards and the surrogate standards may be mixed and added as one spiking solution (this results in a 50 µg/L solution for a 5 mL sample, and a 10 µg/L solution for a 25 mL sample). Inject the sample into the purging chamber.

11.3.4.3.1. For TCLP samples use 0.5 mL of TCLP leachate with 4.5 mL reagent water and spike with 10 µL of the 25 µg/mL TCLP spiking solution.

11.3.4.4. Purge the sample for eleven minutes (the trap must be below 35°C).

11.3.4.5. After purging is complete, desorb the sample, start the GC temperature program, and begin data acquisition. After desorption, bake the trap for 5-10 minutes to condition it for the next analysis. When the trap is cool, it is ready for the next sample.

11.3.4.6. Desorb and bake time and temperature are optimized for the type of trap in use. The same conditions must be used for samples and standards.

11.3.5. Medium-Level Soil/Sediment and Waste Samples

11.3.5.1. Sediments/soils and waste that are insoluble in methanol.

11.3.5.1.1. Gently mix the contents of the sample container with a narrow metal or wood spatula. Weigh 4 g (wet weight) into a tared vial. Use a top-loading balance. Record the weight to 0.1 gram. Do not discard any supernatant liquids.

11.3.5.1.2. Quickly add 9 mL of methanol, and 1 mL of surrogate spiking solution to bring the final volume of methanol to 10 mL. For an LCS or MS/MSD

sample add 8 mL of methanol, 1 mL of surrogate spike solution, and 1 mL of matrix spike solution. Cap the vial and vortex to mix thoroughly.

NOTE: Sections 11.3.5.1.1 and 11.3.5.1.2 must be performed rapidly and without interruption to avoid the loss of volatile organics.

11.3.5.2. Liquid wastes that are soluble in methanol and insoluble in water.

11.3.5.2.1. Pipet 2 mL of the sample into a tared vial. Use a top-loading balance. Record the weight to the nearest 0.1 gram.

11.3.5.2.2. Quickly add 7 mL of methanol, then add 1 mL of surrogate spiking solution to bring the final volume to 10 mL. Cap the vial and shake for 2 minutes to mix thoroughly. For a MS/MSD or LCS, 6 mL of methanol, 1 mL of surrogate solution, and 1 mL of matrix spike solution is used.

11.3.5.3. Fill a 5 mL syringe with 5 mL of reagent water. Add 100 µL (or less if a dilution is required) of methanol extract from the sample preparation in Section 11.3.5.1 or 11.3.5.2. If less than 5 µL of the methanol extract is required, then an intermediate dilution is required. Add 10 µL of the 25 µg/L internal standard solution. (Note that the combined internal standard/surrogate standard solution is not used since surrogates have been added previously.) Inject the sample into the purging chamber and proceed with the analysis as per Sections 11.3.4.4 and 11.3.4.5.

11.3.6. Low-Level Soils

11.3.6.1. This is designed for samples containing individual purgeable compounds of < 2 mg/kg. It is limited to soil/sediment samples and waste that is of a similar consistency (granular and porous). Weigh 5 g of the sample into a tared purge vessel. If a dilution is required, a smaller sample amount can be analyzed, down to a minimum of 1.0 g. Any soil sample requiring further dilution must be run as a medium-level soil. Add 5 mL of reagent water to which 10 µL of the 25 µg/L internal standard/surrogate standard solution has been added. Proceed with the analysis as per Sections 11.3.4.4 and 11.3.4.5.

11.4. Initial review and corrective actions

11.4.1. If the retention time for any internal standard changes by more than 0.5 minutes from the last continuing calibration standard, the chromatographic system must be inspected for malfunctions and corrected. Reanalysis of samples analyzed while the system was malfunctioning is required.

11.4.2. If the retention time of any internal standard in any sample varies by more than 0.1 minute from the preceding continuing calibration standard, the data must be carefully evaluated to ensure that no analytes have shifted outside their retention time windows.

11.4.3. Internal standard response in each sample should be within 50% to 200% of the response in the preceding continuing calibration standard.

11.4.3.1. Any samples that do not meet the internal standard criteria must be evaluated for validity. If the change in sensitivity is a matrix effect confined to an individual sample reanalysis may not be necessary. If the change in sensitivity is due to instrumental problems all affected samples must be reanalyzed after the problem is corrected. In any event, the reason for accepting the sample analysis must be documented. Some clients may require reanalysis of all samples with internal standard criteria outside the 50-200% criteria. Consideration should be given to reanalyzing at a dilution to reduce matrix effects. It is only necessary to reanalyze once to confirm matrix effect.

11.4.4. The surrogate standard recoveries are evaluated to ensure that they are within limits. Corrective action for surrogates out of control will normally be to reanalyze the affected samples. However, if the surrogate standard response is out high and there are no target analytes or tentatively identified compounds, reanalysis may not be necessary. Out of control surrogate standard response may be a matrix effect. It is only necessary to reanalyze a sample once to demonstrate matrix effect, but reanalysis at a dilution should be considered.

11.5. Dilutions

If the response for any compound exceeds the working range of the GC/MS system, a dilution of the extract is prepared and analyzed. An appropriate dilution should be in the upper half of the calibration range. Samples may be screened to determine the appropriate dilution for the initial run. If the initial diluted run has no hits or hits below 20% of the calibration range and the matrix allows for analysis at a lesser dilution, then the sample must be reanalyzed at a dilution targeted to bring the largest hit above 50% of the calibration range.

11.5.1. Guidance for Dilutions Due to Matrix

If the sample is initially run at a dilution and the baseline rise is less than half the height of the peaks in the level 3 standard, then the sample should be reanalyzed at a more concentrated dilution.

11.5.2. Reporting Dilutions

The most concentrated dilution with no target compounds above the calibration range will be reported. Other dilutions will only be reported at client request.

12. DATA ANALYSIS AND CALCULATIONS

12.1. Qualitative identification

An analyte is identified by retention time and by comparison of the sample mass spectrum with the mass spectrum of a standard of the suspected compound (standard reference spectrum). Mass spectra for standard reference may be obtained on the user's GC/MS by analysis of the calibration standards or from the NIST Library. Two criteria must be satisfied to verify identification: (1) elution of sample component at the same GC retention time as the standard component; and (2) correspondence of the sample component and the standard component characteristic ions. (Note: Care must be taken to ensure that spectral distortion due to co-elution is evaluated.)

- The sample component retention time must compare to within ± 0.2 min. of the retention time of the standard component. For reference, the standard must be run within the same twelve hours as the sample.
- All ions present in the standard mass spectra at a relative intensity greater than 10% (most abundant ion in the spectrum equals 100%) should be present in the sample spectrum.
- The relative intensities of ions should agree to within ±30% between the standard and sample spectra. (Example: For an ion with an abundance of 50% in the standard spectra, the corresponding sample abundance must be between 20 and 80 percent.)

12.1.1. If a compound cannot be verified by all the above criteria, but in the technical judgment of the analyst, the identification is correct, then the analyst shall report that identification and proceed with quantitation.

12.2. Tentatively Identified Compounds (TICs)

12.2.1. If the client requests components not associated with the calibration standards, a search of the NIST library may be made for the purpose of tentative identification. Guidelines are:

12.2.1.1. Relative intensities of major ions in the reference spectrum (ions > 10% of the most abundant ion) should be present in the sample spectrum.

12.2.1.2. The relative intensities of the major ions should agree to within 20%. (Example: If an ion shows an abundance of 50% in the standard spectrum, the corresponding sample ion abundance must be between 30% and 70%).

12.2.1.3. Molecular ions present in the reference spectrum should be present in the sample spectrum.

12.2.1.4. Ions present in the sample spectrum but not in the reference spectrum should be reviewed for possible background contamination or presence of coeluting compounds.

12.2.1.5. Ions present in the reference spectrum but not in the sample spectrum should be reviewed for possible subtraction from the spectrum because of background contamination or coeluting peaks. (Data system reduction programs can sometimes create these discrepancies.)

12.2.1.6. Computer-generated library search routines should not use normalization routines that would misrepresent the library or unknown spectra when compared to each other. Only after visual inspection of the sample with the nearest library searches should the analyst assign a tentative identification.

12.3. Calculations.

12.3.1. Response factor (RF):

Equation 1

$$RF = \frac{A_x C_{is}}{A_{is} C_x}$$

Where:

A_x = Area of the characteristic ion for the compound to be measured

A_{is} = Area of the characteristic ion for the specific internal standard

C_{is} = Concentration of the specific internal standard, ng

C_x = Concentration of the compound being measured, ng

12.3.2. Standard deviation (SD):

Equation 2

$$SD = \sqrt{\sum_{i=1}^{N} \frac{(Xi - X)^2}{N - 1}}$$

X_i = Value of X at i through N

N = Number of points

X = Average value of X_i

12.3.3. Percent relative standard deviation (%RSD):

Equation 3

$$\%RSD = \frac{\textbf{Standard Deviation}}{\overline{RF_i}} \times 100$$

$\overline{RF_i}$ = **Mean of RF values in the curve**

12.3.4. Percent drift between the initial calibration and the continuing calibration:

Equation 4

$$\%\ \textbf{Drift} = \frac{C_{expected} - C_{found}}{C_{expected}} \times 100$$

Where

$C_{expected}$ = Known concentration in standard

C_{found} = Measured concentration using selected quantitation method

12.3.5. Target compound and surrogate concentrations:

Concentrations in the sample may be determined from linear or second order (quadratic) curve fitted to the initial calibration points, or from the average response factor of the initial calibration points. Average response factor may only be used when the % RSD of the response factors in the initial calibration is $\leq$ 15%.

12.3.5.1. Calculation of concentration using Quadratic fit

Equation 5

$$\textbf{Concentration } \mu g / L = A + Bx + Cx^2$$

x is defined in equations 8, 9 and 10

A is a constant defined by the intercept

B is the slope of the curve

C is the curvature

12.3.5.2. Calculation of concentration using Linear fit

Equation 6

$$\textbf{Concentration } \mu g / L = A + Bx$$

12.3.5.3. Calculation of concentration using Average Response Factors

Equation 7

$$\textbf{Concentration } \mu g / L = \frac{x}{RF}$$

12.3.5.4. Calculation of x for Water and water-miscible waste:

Equation 8

$$x = \frac{(A_x)(I_s)(D_f)}{(A_{is})(V_o)}$$

Where:

$\mathbf{A_x}$ = Area of characteristic ion for the compound being measured (secondary ion quantitation is allowed only when there are sample interferences with the primary ion)

$\mathbf{A_{is}}$ = Area of the characteristic ion for the internal standard

$\mathbf{I_s}$ = Amount of internal standard added in ng

$$\text{Dilution Factor} = D_f = \frac{\text{Total volume purged (mL)}}{\text{Volume of original sample used (mL)}}$$

$\mathbf{V_o}$ = Volume of water purged, mL

12.3.5.5. Calculation of x for Medium level soils:

Equation 9

$$x = \frac{(A_x)(I_s)(V_t)(1000)(D_f)}{(A_{is})(V_a)(W_s)(D)}$$

Where:

A_x, I_s, D_f, A_{is}, same as for water.

V_t = Volume of total extract, mL (Typically 10 mL)

V_a = Volume of extract added for purging, µL

W_s = Weight of sample extracted, g

$$D = \frac{100 - \%\text{moisture}}{100}$$

12.3.5.6. Calculation of x for Low level soils:

Equation 10

$$x = \frac{(A_x)(I_s)}{(A_{is})(W_s)(D)}$$

Where:

A_x, I_s, A_{is}, same as for water.

D is as for medium level soils

W_s = Weight of sample added to the purge vessel, g

12.3.5.7. Calculation of TICs: The calculation of TICs (tentatively identified compounds) is identical to the above calculations with the following exceptions:

A_x = Area in the total ion chromatogram for the compound being measured

A_{is} = Area of the total ion chromatogram for the nearest internal standard without interference

RF = 1

In other words, the concentration is equal to x as defined in equations 8, 9 and 10.

12.3.6. MS/MSD Recovery

Equation 11

$$\textbf{Matrix Spike Recovery, \%} = \frac{SSR - SR}{SA} \times 100$$

SSR = Spike sample result

SR = Sample result

SA = Spike added

12.3.7. Relative % Difference calculation for the MS/MSD

Equation 12

$$\textbf{RPD} = \frac{|\textbf{MSR} - \textbf{MSDR}|}{\frac{1}{2}(\textbf{MSR} + \textbf{MSDR})} \times 100$$

Where:

RPD = Relative percent difference

MSR = Matrix spike result

MSDR = Matrix spike duplicate result

13. METHOD PERFORMANCE

13.1. Method Detection Limit

Each laboratory must generate a valid method detection limit for each analyte of interest. The MDL must be below the reporting limit for each analyte. The procedure for determination of the method detection limit is given in 40 CFR Part 136, Appendix B, and further defined in QA Policy #: QA-005.

13.2. Initial Demonstration

Each laboratory must make a one time initial demonstration of capability for each individual method. Demonstration of capability for both soil and water matrices is required. This requires analysis of QC check samples containing all of the standard analytes for the method. For some tests it may be necessary to use more than one QC check mix to cover all analytes of interest. The QC check sample is made up at 20 µg/L. (Some compounds will be at higher levels, refer to the calibration standard levels for guidance.)

13.2.1. Four aliquots of the QC check sample are analyzed using the same procedures used to analyze samples, including sample preparation.

13.2.2. Calculate the average recovery and standard deviation of the recovery for each analyte of interest. The %RSD should be $\leq$ 15% for each analyte, and the % recovery should be within 80-120%.

13.2.3. If any analyte does not meet the acceptance criteria, check the acceptance limits in the reference methods (Table 6 of method 8240B, paragraph 8.3.5 of method 8260A). If the recovery or precision is outside the limits in the reference methods, the test must be repeated. Only those analytes that did not meet criteria in the first test need to be evaluated. Repeated failure for any analyte indicates the need for the laboratory to evaluate the analytical procedure and take corrective action.

13.3. Training Qualification

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience.

14. POLLUTION PREVENTION

14.1. This method does not contain any specific modifications that serve to minimize or prevent pollution.

15. WASTE MANAGEMENT

15.1. Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedures. The Health and Safety Director should be contacted if additional information is required.

16. REFERENCES

16.1. SW846, *Test Methods for Evaluating Solid Waste*, Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8240B, Update II, September 1994.

16.2. SW846, *Test Methods for Evaluating Solid Waste,* Third Edition, Gas Chromatography/Mass Spectrometry for Volatile Organics, Method 8260A, Update II, September 1994.

17. MISCELLANEOUS

17.1. Modifications from the reference method

17.1.1. Method 8240B has been modified in this SOP to use capillary columns rather than the packed columns listed in the reference method. This provides improved separation of the target analytes and improved detection limits.

17.1.2. Ion 119 is used as the quantitation ion for chlorobenzene-d5 for 25 mL purge tests.

17.1.3. The internal standard control criteria of 50% to 200% is applied to each sample, rather than the subsequent continuing calibration standard as recommended in the reference method.

17.1.4. A retention time window of 0.2 minutes is used for all components, since some data systems do not have the capability of using the relative retention time units specified in the reference method.

17.1.5. The quantitation and qualifier ions for some compounds have been changed from those recommended in SW-846 in order to improve the reliability of qualitative identification.

17.1.6. Method 8260A recommends that the purge vessel is run through an additional purge cycle after 25 mL sample analysis to remove carryover. Instead, purge vessels are oven baked between analyses or disposable vessels are used one time only.

17.1.7. Matrix spikes and surrogates are spiked at the levels specified in methods 3500 and 5030.

17.1.8. SW-846 recommends that a curve be used for any analytes with %RSD of the response factors > 15%. However, some industry standard data systems and forms generation software cannot report this data with the necessary information for data validation. In addition most software available does not allow weighting of the curve. Unweighted curves may exibit serious errors in quantitation at the low end, resulting in possible false positives or false negatives. Therefore, this SOP allows used of average response factors in 80% of the analytes have %RSDs $\leq$ 15%.Modifications from previous revision

17.1.9. If at least 80% of the analytes in the initial calibration have %RSD < 15%, average response factor calibration may be used far all analytes.

17.2. Facility specific SOPs

Each facility shall attach a list of facility-specific SOPs or approved attachments (if applicable) which are required to implement this SOP or which are used in conjunction with this SOP. If no facility specific SOPs or amendments are to be attached, a statement must be attached specifying that there are none.

Table 2

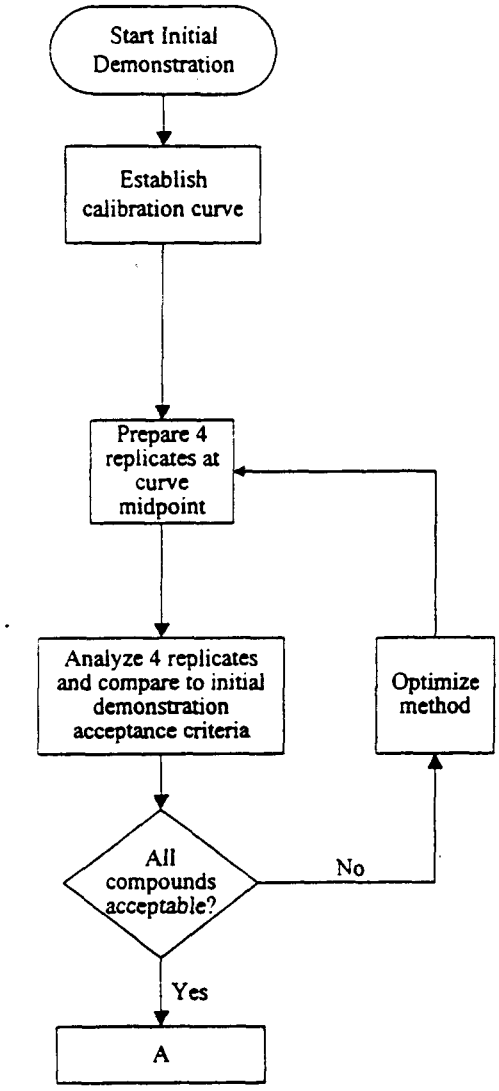
Quanterra Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
Trichloroethene	10	20	50	100	200
Dibromochloromethane	10	20	50	100	200
1,2-Dibromoethane	10	20	50	100	200
1,2,3-Trichloropropane	10	20	50	100	200
1,1,2-Trichloroethane	10	20	50	100	200
Benzene	10	20	50	100	200
Ethylmethacrylate	10	20	50	100	200
trans-1,3-Dichloropropene	10	20	50	100	200
Bromoform	10	20	50	100	200
4-Methyl-2-pentanone	10	20	50	100	200
2-Hexanone	10	20	50	100	200
Tetrachloroethene	10	20	50	100	200
Toluene	10	20	50	100	200
1,1,2,2-Tetrachloroethane	10	20	50	100	200
2-Chloroethyl vinyl ether	20	40	100	200	400
Vinyl acetate	10	20	50	100	200
Chlorobenzene	10	20	50	100	200
Ethylbenzene	10	20	50	100	200
Styrene	10	20	50	100	200
t-1,4-Dichloro-2-butene	10	20	50	100	200
m and p Xylenes	20	40	100	200	400
o-xylene	10	20	50	100	200
1,3-Dichlorobenzene	10	20	50	100	200
1,4-Dichlorobenzene	10	20	50	100	200
1,2-Dichlorobenzene	10	20	50	100	200

[1] **Levels for 25 mL purge are 5 times lower in all cases**

17.3. Flow diagrams

17.3.1. Initial Demonstration and MDL



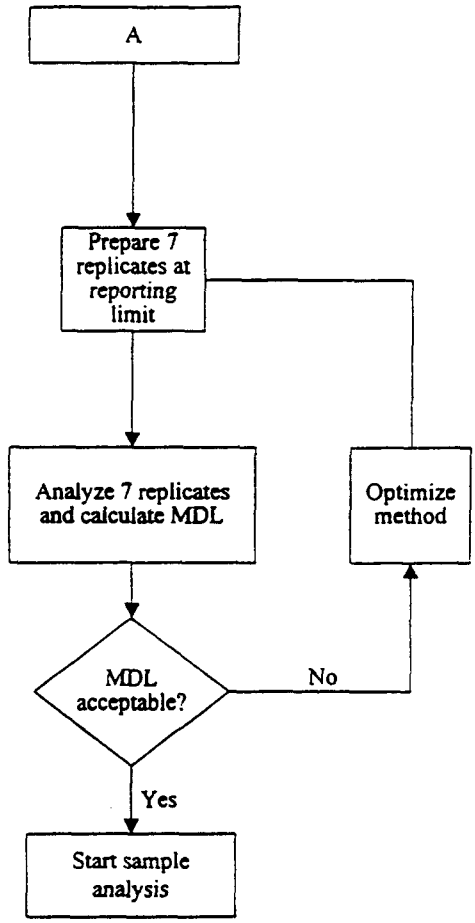


Table 1

Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	1200
Chloromethane	74-87-3	10	2	10	1200
Bromomethane	74-83-9	10	2	10	1200
Vinyl chloride	75-01-4	10	2	10	1200
Chloroethane	75-00-3	10	2	10	1200
Trichlorofluoromethane	75-69-4	10	2	10	1200
Acrolein	107-02-8	100	20	100	12000
Acetone	67-64-1	20	10	20	2500
Trichlorotrifluoroethane	76-13-1	5	1	5	620
Ethanol	64-17-5	500	200	500	62,000
Iodomethane	74-88-4	5	1	5	620
Carbon disulfide	75-15-0	5	1	5	620
Methylene chloride	75-09-2	5	1	5	620
tert-Butyl alcohol	75-65-0	200	50	200	25,000
1,1-Dichloroethene	75-35-4	5	1	5	620
1,1-Dichloroethane	75-34-3	5	1	5	620
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	310
Acrylonitrile	107-13-1	100	20	100	12000
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	2500
Hexane	110-54-3	5	1	5	620
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	620
Tetrahydrofuran	109-99-9	20	5	20	2500
Chloroform	67-66-3	5	1	5	620
1,2-Dichloroethane	107-06-2	5	1	5	620
Dibromomethane	74-95-3	5	1	5	620
2-Butanone	78-93-3	20	5	20	2500
1,4-Dioxane	123-91-1	500	200	500	62000
1,1,1-Trichloroethane	71-55-6	5	1	5	620
Carbon tetrachloride	56-23-5	5	1	5	620
Bromodichloromethane	75-27-4	5	1	5	620
1,2-Dichloropropane	78-87-5	5	1	5	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	620
Trichloroethene	79-01-6	5	1	5	620

Table 1

Quanterra Primary Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL Water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dibromochloromethane	124-48-1	5	1	5	620
1,2-Dibromoethane	106-93-4	5	1	5	620
1,2,3-Trichloropropane	96-18-4	5	1	5	620
1,1,2-Trichloroethane	79-00-5	5	1	5	620
Benzene	71-43-2	5	1	5	620
Ethylmethacrylate	97-63-2	5	1	5	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	620
Bromoform	75-25-2	5	1	5	620
4-Methyl-2-pentanone	108-10-1	20	5	20	2500
2-Hexanone	591-78-6	20	5	20	2500
Tetrachloroethene	127-18-4	5	1	5	620
Toluene	108-88-3	5	1	5	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	620
2-Chloroethyl vinyl ether	110-75-8	N/A[2]	N/A	50	6200
Vinyl acetate	108-05-4	10	2	10	1200
Chlorobenzene	108-90-7	5	1	5	620
Ethylbenzene	100-41-4	5	1	5	620
Styrene	100-42-5	5	1	5	620
t-1,4-Dichloro-2-butene	110-57-6	5	1	5	620
m and p Xylenes		2.5	0.5	2.5	310
o-xylene	95-47-6	2.5	0.5	2.5	310
Total xylenes	1330-20-7	5	1	5	620
1,3-Dichlorobenzene	541-73-1	5	1	5	620
1,4-Dichlorobenzene	106-46-7	5	1	5	620
1,2-Dichlorobenzene	95-50-1	5	1	5	620

[1] Reporting limits listed for soil/sediment are based on wet weight. The reporting limits calculated by the laboratory for soil/sediment calculated on dry weight basis, will be higher.

[2] 2-Chloroethyl vinyl ether cannot be reliably recovered from acid preserved samples

Table 2

Quanterra Primary Standard Calibration Levels, 5 mL purge[1]

Compound	Calibration Level ug/L				
	Level 1	Level 2	Level 3	Level 4	Level 5
1,2-Dichloroethane-d4 (Surrogate)	10	20	50	100	200
Toluene-d8 (Surrogate)	10	20	50	100	200
4-Bromofluorobenzene (Surrogate)	10	20	50	100	200
Dichlorodifluoromethane	10	20	50	100	200
Chloromethane	10	20	50	100	200
Bromomethane	10	20	50	100	200
Vinyl chloride	10	20	50	100	200
Chloroethane	10	20	50	100	200
Trichlorofluoromethane	10	20	50	100	200
Acrolein	100	200	500	1000	2000
Acetone	10	20	50	100	200
Trichlorotrifluoroethane	10	20	50	100	200
Ethanol	500	1000	5000	10,000	20,000
Iodomethane	10	20	50	100	200
Carbon disulfide	10	20	50	100	200
Methylene chloride	10	20	50	100	200
tert-Butyl alcohol	200	400	1,000	2,000	4,000
1,1-Dichloroethene	10	20	50	100	200
1,1-Dichloroethane	10	20	50	100	200
trans-1,2-Dichloroethene	10	20	50	100	200
Acrylonitrile	100	200	500	1,000	2,000
Methyl *tert*-butyl ether (MTBE)	10	20	50	100	200
Hexane	10	20	50	100	200
cis-1,2-Dichloroethene	10	20	50	100	200
Tetrahydrofuran	10	20	50	100	200
Chloroform	10	20	50	100	200
1,2-Dichloroethane	10	20	50	100	200
Dibromomethane	10	20	50	100	200
2-Butanone	10	20	50	100	200
1,4-Dioxane	500	1000	2,500	5,000	10,000
1,1,1-Trichloroethane	10	20	50	100	200
Carbon tetrachloride	10	20	50	100	200
Bromodichloromethane	10	20	50	100	200
1,2-Dichloropropane	10	20	50	100	200
cis-1,3-Dichloropropene	10	20	50	100	200

Table 3

Quanterra Appendix IX Standard and Reporting Limits, 5 mL purge[1]

Compound	CAS Number	Reporting Limits			
		5 mL Water µg/L	25 mL water µg/L	Low Soil µg/kg	Medium Soil µg/mL
Allyl Chloride	107-05-1	10	2	10	1,200
Acetonitrile	75-05-8	100	20	100	12,000
Dichlorofluoromethane		10	2	10	1,200
Isopropyl ether	108-20-3	50	10	50	6,200
Chloroprene	126-99-8	5	1	5	620
n-Butanol	71-36-3	200	50	200	25,000
Propionitrile	107-12-0	20	4	20	2500
Methacrylonitrile	126-98-7	5	1	5	620
Isobutanol	78-83-1	200	50	200	25,000
Methyl methacrylate	80-62-6	5	1	5	620
1,1,1,2-Tetrachloroethane	630-20-6	5	1	5	620
1,2-Dibromo-3-chloropropane	96-12-8	10	2	10	1,200
Ethyl ether	60-29-7	10	2	10	1,200
Ethyl Acetate	141-78-6	20	4	20	2500
2-Nitropropane	79-46-9	10	2	10	1,200
Cyclohexanone	108-94-1	N/A[2]	N/A[2]	N/A[2]	N/A[2]
Isopropylbenzene	98-82-8	5	1	5	620

[1] **Levels for 25 mL purge are 5 times lower in all cases**

[2] Cyclohexanone decomposes to 1,1-dimethoxycyclohexane in methanolic solution. Reporting limits cannot be accurately determined.

Table 4

Quanterra Appendix IX Standard Calibration Levels, µg/L

Compound	Level 1	Level 2	Level 3	Level 4	Level 5
Allyl Chloride	10	20	50	100	200
Acetonitrile	100	200	500	1,000	2,000
Dichlorofluoromethane	10	20	50	100	200
Isopropyl ether	50	100	250	500	1,000
Chloroprene	10	20	50	100	200
n-Butanol	200	400	1,000	2,000	4,000
Propionitrile	20	40	100	200	400
Methacrylonitrile	10	20	50	100	200
Isobutanol	200	400	1,000	2,000	4,000
Methyl methacrylate	10	20	50	100	200
1,1,1,2-Tetrachloroethane	10	20	50	100	200
1,2-Dibromo-3-chloropropane	20	40	100	200	400
Ethyl ether	10	20	50	100	200
Ethyl Acetate	20	40	100	200	400
2-Nitropropane	20	40	100	200	400
Cyclohexanone	100	200	500	1,000	2,000
Isopropylbenzene	10	20	50	100	200

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Dichlorodifluoromethane	75-71-8				X	X
Chloromethane	74-87-3	X		X	X	X
Bromomethane	74-83-9	X		X	X	X
Vinyl chloride	75-01-4	X	X	X	X	X
Chloroethane	75-00-3	X		X	X	X
Trichlorofluoromethane	75-69-4				X	X
Acrolein	107-02-8				X	X
Acetone	67-64-1	X		X	X	X
Trichlorotrifluoroethane	76-13-1					X
Ethanol	64-17-5					
Iodomethane	74-88-4				X	X
Carbon disulfide	75-15-0	X		X	X	X
Methylene chloride	75-09-2	X		X	X	X
tert-Butly alcohol	75-65-0					
1,1-Dichloroethene	75-35-4	X	X	X	X	X
1,1-Dichloroethane	75-34-3	X		X	X	X
trans-1,2-Dichloroethene	156-60-5	X		X	X	X
Total dichloroethene		X		X	X	X
Acrylonitrile	107-13-1					X
Methyl *tert*-butyl ether (MTBE)	1634-04-4					
Hexane	110-54-3					
cis-1,2-Dichloroethene	156-59-2	X		X		
Tetrahydrofuran	109-99-9					
Chloroform	67-66-3	X	X	X	X	X
1,2-Dichloroethane	107-06-2	X	X	X	X	X
Dibromomethane	74-95-3				X	X
2-Butanone	78-93-3	X	X	X	X	X
1,4-Dioxane	123-91-1				X	X
1,1,1-Trichloroethane	71-55-6	X		X	X	X
Carbon tetrachloride	56-23-5	X	X	X	X	X
Bromodichloromethane	75-27-4	X		X	X	X
1,2-Dichloropropane	78-87-5	X		X	X	X
cis-1,3-Dichloropropene	10061-01-	X		X	X	X

Table 5

Reportable Analytes for Quanterra Standard Tests, Primary Standard

Compound	CAS Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
	5					
Trichloroethene	79-01-6	X	X	X	X	X
Dibromochloromethane	124-48-1	X		X	X	X
1,2-Dibromoethane	106-93-4				X	X
1,2,3-Trichloropropane	96-18-4				X	X
1,1,2-Trichloroethane	79-00-5	X		X	X	X
Benzene	71-43-2	X	X	X	X	X
Ethylmethacrylate	97-63-2				X	X
trans-1,3-Dichloropropene	10061-02-6	X		X	X	X
Bromoform	75-25-2	X		X	X	X
4-Methyl-2-pentanone	108-10-1	X		X	X	X
2-Hexanone	591-78-6	X		X	X	
Tetrachloroethene	127-18-4	X	X	X	X	X
Toluene	108-88-3	X		X	X	X
1,1,2,2-Tetrachloroethane	79-34-5	X		X	X	X
2-Chloroethyl vinyl ether	110-75-8					
Vinyl acetate	108-05-4				X	
Chlorobenzene	108-90-7	X	X	X	X	X
Ethylbenzene	100-41-4	X		X	X	X
Styrene	100-42-5	X		X	X	
t-1,4-Dichloro-2-butene	110-57-6				X	
m and p Xylenes		X		X	X	X
o-xylene	95-47-6	X		X	X	X
Total xylenes	1330-20-7	X		X	X	X
1,3-Dichlorobenzene	541-73-1					
1,4-Dichlorobenzene	106-46-7					
1,2-Dichlorobenzene	95-50-1					

Table 6

Reportable Analytes for Quanterra Standard Tests, Appendix IX standard

Compound	Number	Quanterra Standard List	TCLP	TCL	Appendix IX	UTS
Allyl Chloride	107-05-1				X	
Acetonitrile	75-05-8				X	X
Dichlorofluoromethane	75-43-4					
Isopropyl ether	108-20-3					
Chloroprene	126-99-8				X	
n-Butanol	71-36-3					
Propionitrile	107-12-0				X	
Methacrylonitrile	126-98-7				X	X
Isobutanol	78-83-1				X	X
Methyl methacrylate	80-62-6				X	X
1,1,1,2-Tetrachloroethane	630-20-6				X	X
1,2-Dibromo-3-chloropropane	96-12-8				X	X
Ethyl ether	60-29-7					X
Ethyl Acetate	141-78-6					X
2-Nitropropane	79-46-9					
Cyclohexanone	108-94-1					X
Isopropylbenzene	98-82-8					

Table 7

Internal Standards

	Standard Concentration µg/mL	Quantitation ion (5 mL purge)	Quantitation ion (25 mL purge)
Bromochloromethane	25	128	128
1,4-Difluorobenzene	25	114	114
Chlorobenzene d5	25	117	119

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 8

Surrogate Standards

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

3) Recovery limits for surrogates are generated from historical data and are maintained by the QA department.

Table 9

Matrix Spike / LCS Compounds

Compound	Standard Concentration µg /mL
1,1-Dichloroethene	25
Trichloroethene	25
Toluene	25
Benzene	25
Chlorobenzene	25

Notes:

1) 10 µL of the standard is added to the LCS or matrix spiked sample. This results in a concentration of each spike analyte in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Recovery and precision limits for LCS and MS/MSD are generated from historical data and are maintained by the QA dedartment.

Table 10

BFB Key Ion Abundance Criteria

Mass	Ion Abundance Criteria
50	15% to 40% of Mass 95
75	30% to 60% of Mass 95
95	Base Peak, 100% Relative Abundance
96	5% to 9% of Mass 95
173	Less Than 2% of Mass 174
174	Greater Than 50% of Mass 95
175	5% to 9% of Mass 174
176	Greater Than 95%, But Less Than 101% of Mass 174
177	5% to 9% of Mass 176

Table 11

SPCC Compounds and Minimum Response Factors

Compound	8240B Min. RF	8260A Min. RF
Chloromethane	0.300	0.100
1,1-Dichloroethane	0.300	0.100
Bromoform	>0.100	>0.100
1,1,2,2-Tetrachloroethane	0.300	0.300
Chlorobenzene	0.300	0.300

Table 12

CCC compounds

Compound	Max. %RSD from Initial Calibration	Max. %D for continuing calibration
Vinyl Chloride	<30.0	<20.0
1,1-Dichloroethene	<30.0	<20.0
Chloroform	<30.0	<20.0
1,2-Dichloropropane	<30.0	<20.0
Toluene	<30.0	<20.0
Ethylbenzene	<30.0	<20.0

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Bromochloromethane (Internal Standard)	128	49	130, 51
1,2-Dichloroethane-d_4 (Surrogate)	65	102	
Dichlorodifluoromethane	85	87	50, 101,103
Chloromethane	50	52	49
Vinyl chloride	62	64	61
Bromomethane	94	96	79
Chloroethane	64	66	49
Trichlorofluoromethane	101	103	66
1,1-Dichloroethene	96	61	98
Acrolein	56	55	58

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
Iodomethane	142	127	141
Carbon disulfide	76	78	
Trichlorotrifluoroethane	151	101	153
Ethanol	45	46	
Acetone	43	58	
Methylene chloride	84	49	51, 86
tert-Butyl alcohol	59	74	
trans-1,2-Dichloroethene	96	61	98
Acrylonitrile	53	52	51
Methyl *tert* butyl ether	73		
Hexane	57	43	
1,1-Dichloroethane	63	65	83
cis-1,2-Dichloroethene	96	61	98
2-Butanone	43	72**	
Tetrahydrofuran	42	71	
Chloroform	83	85	47
1,2-Dichloroethane	62	64	98
Dibromomethane	93	174	95, 172, 176
1,4-Dioxane	88	58	
1,4-Difluorobenzene (Internal Standard)	114	63	88
Vinyl acetate	43	86	
1,1,1-Trichloroethane	97	99	117
Carbon tetrachloride	117	119	121
Benzene	78	52	77
Trichloroethene	130	95	97, 132
1,2-Dichloropropane	63	65	41
Bromodichloromethane	83	85	129
2-Chloroethyl vinyl ether	63	65	106
cis-1,3-Dichloropropene	75	77	39
trans-1,3-Dichloropropene	75	77	39
1,1,2-Trichloroethane	97	83	85, 99
Chlorodibromomethane	129	127	131
Bromoform	173	171	175, 252
1,2,3-Trichloropropane	75	110	77, 112, 97
Chlorobenzene-d_5 (Internal Standard)	117,119	117,119	
Toluene-d_8 (Surrogate)	98	70	100
4-Bromofluorobenzene (Surrogate)	95	174	176
Toluene	91	92	65

Table 13

Characteristic ions

Compound	Primary*	Secondary	Tertiary
4-Methyl-2-pentanone	43	58	57, 100
Tetrachloroethene	164	166	131
Ethyl methacrylate	69	41	99, 86, 114
2-Hexanone	43	58	57, 100
Chlorobenzene	112	114	77
Ethylbenzene	106	91	
Xylenes	106	91	
Styrene	104	103	78, 51, 77
Dichlorobenzene (all isomers)	146	148	111
trans 1,4-Dichloro-2-butene	53	75	89, 77, 124
1,1,2,2-Tetrachloroethane	83	85	131, 133
Allyl Chloride	76	41	78
Acetonitrile	40	41	
Dichlorofluoromethane	67	69	
Isopropyl ether	87	59	45
Chloroprene	53	88	90
n-Butanol	56	41	42
Propionitrile	54	52	55
Methacrylonitrile	41	67	52
Isobutanol	41	43	74
Methyl methacrylate	41	69	100
1,1,1,2-Tetrachloroethane	131	133	119
1,2-Dibromo-3-chloropropane	157	155	75
Ethyl ether	59	74	
Ethyl Acetate	43	88	61
2-Nitropropane	41	43	46
Cyclohexanone	55	42	98
Isopropylbenzene	105	120	

* The primary ion should be used for quantitation unless interferences are present, in which case a secondary ion may be used.

** m/z 43 may be used for quantitation of 2-Butanone, but m/z 72 must be present for positive identification.

1. SUMMARY

A target analyte list based on the list in method 524.2 is frequently requested for analysis by method 8260A. Quanterra's standard analyte list for this test, and the internal and surrogate standards used, are listed in Tables below.. In all other respects the method is as described in the main body of this SOP. Note that this SOP is ***not*** appropriate for drinking water analysis by method 524.2.

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Dichlorodifluoromethane	75-71-8	10	2	10	1200
Chloromethane	74-87-3	10	2	10	1200
Bromomethane	74-83-9	10	2	10	1200
Vinyl chloride	75-01-4	10	2	10	1200
Chloroethane	75-00-3	10	2	10	1200
Trichlorofluoromethane	75-69-4	10	2	10	1200
Acetone[1]	67-64-1	20	10	20	2500
Methylene chloride	75-09-2	5	2	5	620
1,1-Dichloroethene	75-35-4	5	1	5	620
1,1-Dichloroethane	75-34-3	5	1	5	620
trans-1,2-Dichloroethene	156-60-5	2.5	0.5	2.5	310
Methyl *tert*-butyl ether (MTBE)	1634-04-4	20	5	20	620
2,2-Dichloropropane	590-20-7	5	1	5	620
cis-1,2-Dichloroethene	156-59-2	2.5	0.5	2.5	310
1,2-Dichloroethene (Total)	540-59-0	5	1	5	620
Chloroform	67-66-3	5	1	5	620
Bromochloromethane	74-97-5	5	1	5	620
1,2-Dichloroethane	107-06-2	5	1	5	620
Dibromomethane	74-95-3	5	1	5	620
2-Butanone[1]	78-93-3	20	5	20	2500
1,1,1-Trichloroethane	71-55-6	5	1	5	620
Carbon tetrachloride	56-23-5	5	1	5	620
Bromodichloromethane	75-27-4	5	1	5	620
1,2-Dichloropropane	78-87-5	5	1	5	620
cis-1,3-Dichloropropene	10061-01-5	5	1	5	620

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Trichloroethene	79-01-6	5	1	5	620
Dibromochloromethane	124-48-1	5	1	5	620
1,2-Dibromoethane	106-93-4	5	1	5	620
1,2,3-Trichloropropane	96-18-4	5	1	5	620
1,1,2-Trichloroethane	79-00-5	5	1	5	620
Benzene	71-43-2	5	1	5	620
trans-1,3-Dichloropropene	10061-02-6	5	1	5	620
Bromoform	75-25-2	5	1	5	620
4-Methyl-2-pentanone[1]	108-10-1	20	5	20	2500
2-Hexanone[1]	591-78-6	20	5	20	2500
Tetrachloroethene	127-18-4	5	1	5	620
Toluene	108-88-3	5	1	5	620
1,1,2,2-Tetrachloroethane	79-34-5	5	1	5	620
Chlorobenzene	108-90-7	5	1	5	620
Ethylbenzene	100-41-4	5	1	5	620
Styrene	100-42-5	5	1	5	620
m and p Xylenes		2.5	0.5	2.5	310
o-xylene	95-47-6	2.5	0.5	2.5	310
Total xylenes	1330-20-7	5	1	5	620
Isopropylbenzene	98-82-8	5	1	5	620
Bromobenzene	108-86-1	5	1	5	620
n-Propylbenzene	103-65-1	5	1	5	620
2-Chlorotoluene	95-49-8	5	1	5	620
4-Chlorotoluene	106-43-4	5	1	5	620
1,3,5-Trimethylbenzene	108-67-8	5	1	5	620
tert-Butylbenzene	98-06-6	5	1	5	620
1,2,4-Trimethylbenzene	95-63-6	5	1	5	620
sec-butylbenzene	135-98-8	5	1	5	620
1,3-Dichlorobenzene	541-73-1	5	1	5	620
1,4-Dichlorobenzene	106-46-7	5	1	5	620
1,2-Dichlorobenzene	95-50-1	5	1	5	620
4-Isopropyltoluene	99-87-6	5	1	5	620
n-Butylbenzene	104-51-8	5	1	5	620
1,2-Dibromo-3-chloropropane	96-12-8	5	1	5	620
1,2,4-Trichlorobenzene	120-82-1	5	1	5	620

Table 14

Quanterra 8260 Drinking Water List Standard and Reporting Limits

Compound	CAS Number	Reporting Limits[1]			
		5 mL water µg/L	25 mL water µg/L	Low soil µg/kg	Med. Soil µg/kg
Napthalene	91-20-3	5	1	5	620
Hexachlorobutadiene	87-68-3	5	1	5	620
1,2,3-Trichlorobenzene	87-61-6	5	1	5	620

[1] Not included on the method 524.2 analyte list, but includes in the calibration standard as an add on frequently requested by method 8260A.

Table 15

Internal Standards, Method 8260A Drinking water list

	Standard Concentration µg/mL	Quantitation ion
Fluorobenzene	25	128
Chlorobenzene-d5	25	114
1,4-Dichlorobenzene-d4	25	119

Notes:

1) 10 µL of the internal standard is added to the sample. This results in a concentration of each internal in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 16

Surrogate Standards, Method 8260B Drinking water list

Surrogate Compounds	Standard Concentration µg/mL
1,2-Dichloroethane-d_4	25
Toluene-d_8	25
4-Bromofluorobenzene	25

Notes:

1) 10 µL of the surrogate standard is added to the sample. This results in a concentration of each surrogate in the sample of 50µg/L for a 5 mL purge or 10 µg/L for a 25 mL purge.

2) Except for medium level soils, the surrogate and internal standards may be combined in one solution.

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL	5 mL	25 mL
Dichlorodifluoromethane	20	4	40	10	100	20	200	60	400	120
Chloromethane	20	4	40	10	100	20	200	60	400	120
Bromomethane	20	4	40	10	100	20	200	60	400	120
Vinyl chloride	20	4	40	10	100	20	200	60	400	120
Chloroethane	20	4	40	10	100	20	200	60	400	120
Trichlorofluoromethane	20	4	40	10	100	20	200	60	400	120
Acetone[1]	20	4	40	10	100	20	200	60	400	120
Methylene chloride	10	2	20	5	50	10	100	30	200	60
1,1-Dichloroethene	10	2	20	5	50	10	100	30	200	60
1,1-Dichloroethane	10	2	20	5	50	10	100	30	200	60
trans-1,2-Dichloroethene	10	2	20	5	50	10	100	30	200	60
Methyl *tert*-butyl ether (MTBE)	20	4	40	10	100	20	200	60	400	120
2,2-Dichloropropane	10	2	20	5	50	10	100	30	200	60
cis-1,2-Dichloroethene	10	2	20	5	50	10	100	30	200	60

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Chloroform	10	2	20	5	50	10	100	30	200	60
Bromochloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dichloroethane	10	2	20	5	50	10	100	30	200	60
Dibromomethane	10	2	20	5	50	10	100	30	200	60
2-Butanone[1]	20	4	40	10	100	20	200	60	400	120
1,1,1-Trichloroethane	10	2	20	5	50	10	100	30	200	60
Carbon tetrachloride	10	2	20	5	50	10	100	30	200	60
Bromodichloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dichloropropane	10	2	20	5	50	10	100	30	200	60
cis-1,3-Dichloropropene	10	2	20	5	50	10	100	30	200	60
Trichloroethene	10	2	20	5	50	10	100	30	200	60
Dibromochloromethane	10	2	20	5	50	10	100	30	200	60
1,2-Dibromoethane	10	2	20	5	50	10	100	30	200	60
1,2,3-Trichloropropane	10	2	20	5	50	10	100	30	200	60
1,1,2-Trichloroethane	10	2	20	5	50	10	100	30	200	60
Benzene	10	2	20	5	50	10	100	30	200	60
trans-1,3-Dichloropropene	10	2	20	5	50	10	100	30	200	60
Bromoform	10	2	20	5	50	10	100	30	200	60
4-Methyl-2-pentanone[1]	20	4	40	10	100	20	200	60	400	120
2-Hexanone[1]	20	4	40	10	100	20	200	60	400	120
Tetrachloroethene	10	2	20	5	50	10	100	30	200	60
Toluene	10	2	20	5	50	10	100	30	200	60
1,1,2,2-Tetrachloroethane	10	2	20	5	50	10	100	30	200	60
Chlorobenzene	10	2	20	5	50	10	100	30	200	60
Ethylbenzene	10	2	20	5	50	10	100	30	200	60
Styrene	10	2	20	5	50	10	100	30	200	60
m and p Xylenes	10	2	20	5	50	10	100	30	200	60
o-xylene	10	2	20	5	50	10	100	30	200	60

Table 17

Quanterra 8260 Drinking water list Standard: Calibration Levels

Compound	Level 1		Level 2		Level 3		Level 4		Level 5	
Isopropylbenzene	10	2	20	5	50	10	100	30	200	60
Bromobenzene	10	2	20	5	50	10	100	30	200	60
n-Propylbenzene	10	2	20	5	50	10	100	30	200	60
2-Chlorotoluene	10	2	20	5	50	10	100	30	200	60
4-Chlorotoluene	10	2	20	5	50	10	100	30	200	60
1,3,5-Trimethylbenzene	10	2	20	5	50	10	100	30	200	60
tert-Butylbenzene	10	2	20	5	50	10	100	30	200	60
1,2,4-Trimethylbenzene	10	2	20	5	50	10	100	30	200	60
sec-butylbenzene	10	2	20	5	50	10	100	30	200	60
1,3-Dichlorobenzene	10	2	20	5	50	10	100	30	200	60
1,4-Dichlorobenzene	10	2	20	5	50	10	100	30	200	60
1,2-Dichlorobenzene	10	2	20	5	50	10	100	30	200	60
4-Isopropyltoluene	10	2	20	5	50	10	100	30	200	60
n-Butylbenzene	10	2	20	5	50	10	100	30	200	60
1,2-Dibromo-3-chloropropane	10	2	20	5	50	10	100	30	200	60
1,2,4-Trichlorobenzene	10	2	20	5	50	10	100	30	200	60
Napthalene	10	2	20	5	50	10	100	30	200	60
Hexachlorobutadiene	10	2	20	5	50	10	100	30	200	60
1,2,3-Trichlorobenzene	10	2	20	5	50	10	100	30	200	60

[1] Not included in the Quanterra Standard test, but included in the standard as a frequently requested add-on.

APPENDIX B-11

STANDARD TEST METHOD FOR SULFUR IN PETROLEUM PRODUCTS (GENERAL BOMB METHOD)

Designation: D 129 – 91

An American National Standard
British Standard 4454

Designation: 61/84

Standard Test Method for Sulfur in Petroleum Products (General Bomb Method)[1]

This standard is issued under the fixed designation D 129; the number immediately following the designation indicates the year of original adoption or, in the case of revision, the year of last revision. A number in parentheses indicates the year of last reapproval. A superscript epsilon (ε) indicates an editorial change since the last revision or reapproval.

This test method was adopted as a joint ASTM-IP standard in 1964.

This test method has been adopted for use by government agencies to replace Method 5202 of Federal Test Method Standard No. 791b.

Attention is called to Section 3.2 on Safety and to the specific precautionary directions incorporated in the test method.

1. Scope

1.1 This test method covers the determination of sulfur in petroleum products, including lubricating oils containing additives, additive concentrates, and lubricating greases, that cannot be burned completely in a wick lamp. The test method is applicable to any petroleum product sufficiently low in volatility that it can be weighed accurately in an open sample boat and containing at least 0.1 % sulfur.

NOTE 1—This test method is not applicable to samples containing elements that give residues, other than barium sulfate, which are insoluble in dilute hydrochloric acid and would interfere in the precipitation step. These interfering elements include iron, aluminum, calcium, silicon, and lead which are sometimes present in greases, lube oil additives, or additive oils. Other acid insoluble materials that interfere are silica, molybdenum disulfide, asbestos, mica, etc. The test method is not applicable to used oils containing wear metals, and lead or silicates from contamination. Samples that are excluded can be analyzed by Test Method D 1552.

2. Referenced Document

2.1 *ASTM Standard:*

D 1552 Test Method for Sulfur in Petroleum Products (High-Temperature Method)[2]

3. Summary of Test Method

3.1 The sample is oxidized by combustion in a bomb containing oxygen under pressure. The sulfur, as sulfate in the bomb washings, is determined gravimetrically as barium sulfate.

3.2 **Warning**—*Strict adherence to all of the provisions prescribed hereafter ensures against explosive rupture of the bomb, or a blow-out, provided the bomb is of proper design and construction and in good mechanical condition. It is desirable, however, that the bomb be enclosed in a shield of steel plate at least 13 mm thick, or equivalent protection be provided against unforseeable contingencies.*

4. Apparatus and Materials

4.1 *Bomb*,[3,4] having a capacity of not less than 300 mL, so constructed that it will not leak during the test and that quantitative recovery of the liquids from the bomb may be achieved readily. The inner surface of the bomb may be made of stainless steel or any other material that will not be affected by the combustion process or products. Materials used in the bomb assembly, such as the head gasket and lead-wire insulation, shall be resistant to heat and chemical action, and shall not undergo any reaction that will affect the sulfur content of the liquid in the bomb.

4.2 *Sample Cup*, platinum, 24 mm in outside diameter at the bottom, 27 mm in outside diameter at the top, 12 mm in height outside, and weighing 10 to 11 g.

4.3 *Firing Wire*, platinum, approximately No. 26 B & S gage, 27 SWG, or equivalent.

NOTE 2: **Caution**—The switch in the ignition circuit shall be of a type which remains open, except when held in closed position by the operator.

4.4 *Ignition Circuit*, capable of supplying sufficient current to ignite the cotton wicking or nylon thread without melting the wire. The current shall be drawn from a step-down transformer or from a suitable battery.

4.5 *Cotton Wicking or Nylon Sewing Thread*, white.

5. Reagents and Materials

5.1 *Purity of Reagents*—Reagent grade chemicals shall be used in all tests. Unless otherwise indicated, it is intended that all reagents shall conform to the specifications of the Committee on Analytical Reagents of the American Chemical Society, where such specifications are available.[5] Other grades may be used, provided it is first ascertained that the reagent is of sufficiently high purity to permit its use without lessening the accuracy of the determination.

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[1] This test method is under the jurisdiction of ASTM Committee D-2 on Petroleum Products and Lubricants and is the direct responsibility of Subcommittee D02.03 on Elemental Analysis.

Current edition approved Oct. 15, 1991. Published December 1991. Originally published as D 129 – 22. Last previous edition D 129 – 64 (Reapproved 1978).[ε1]

In the IP, this test method is under the jurisdiction of the Standardization Committee.

[2] *Annual Book of ASTM Standards*, Vol 05.01.

[3] Criteria for judging the acceptability of new and used oxygen combustion bombs are described in Practice E 144, *Annual Book of ASTM Standards*, Vol 14.02.

[4] A bomb conforming to the test specifications in IP Standard IP 12 is suitable.

[5] "Reagent Chemicals, American Chemical Society Specifications," Am. Chemical Soc., Washington, D.C. For suggestions on the testing of reagents not listed by the American Chemical Society, see "Reagent Chemicals and Standards," by Joseph Rosin, D. Van Nostrand Co., Inc., New York, N. Y., and the "United States Pharmacopeia."

5.2 *Purity of Water*—Unless otherwise indicated, references to water shall be understood to mean distilled water or water of equal purity.

5.3 *Barium Chloride Solution* (85 g/litre)—Dissolve 100 g of barium chloride dihydrate ($BaCl_2 \cdot 2H_2O$) in distilled water and dilute to 1 liter.

5.4 *Bromine Water (saturated).*

5.5 *Hydrochloric Acid* (sp gr 1.19)—Concentrated hydrochloric acid (HCl).

5.6 *Oxygen*, free of combustible material and sulfur compounds, available at a pressure of 40 atm (41 kgf/cm²).

5.7 *Sodium Carbonate Solution* (50 g/litre)—Dissolve 135 g of sodium carbonate decahydrate ($Na_2CO_3 \cdot 10H_2O$) or its equivalent weight in distilled water and dilute to 1 litre.

5.8 *White Oil, USP, or Liquid Paraffin.* BP, *or equivalent.*

6. Procedure

6.1 *Preparation of Bomb and Sample*—Cut a piece of firing wire 100 mm in length. Coil the middle section (about 20 mm) and attach the free ends to the terminals. Arrange the coil so that it will be above and to one side of the sample cup. Insert between two loops of the coil a wisp of cotton or nylon thread of such length that one end will extend into the sample cup. Place about 5 mL of Na_2CO_3 solution in the bomb (Note 3) and rotate the bomb in such a manner that the interior surface is moistened by the solution. Introduce into the sample cup the quantities of sample and white oil (Notes 5 and 6) specified in the following table, weighing the sample to the nearest 0.2 mg (when white oil is used, stir the mixture with a short length of quartz rod and allow the rod to remain in the sample cup during the combustion).

NOTE 3—After repeated use of the bomb for sulfur determinations, a film may be noticed on the inner surface. This dullness should be removed by periodic polishing of the bomb. A satisfactory method for doing this is to rotate the bomb in a lathe at about 300 rpm and polish the inside surface with emery polishing papers Grit No. 3/0, or equivalent paper,[6] coated with a light machine oil to prevent cutting, and then with a paste of grit-free chromic oxide[3] and water. This procedure will remove all but very deep pits and put a high polish on the surface. Before the bomb is used it should be washed with soap and water to remove oil or paste left from the polishing operation.

NOTE 4: Caution—Do not use more than 1.0 g total of sample and white oil or other low sulfur combustible material or more than 0.8 g if the IP 12 bomb is used.

Sulfur Content percent	Weight of Sample, g	Weight of White Oil, g
5 or under	0.6 to 0.8	0.0
Over 5	0.3 to 0.4	0.3 to 0.4

NOTE 5—Use of sample weights containing over 20 mg of chlorine may cause corrosion of the bomb. To avoid this, it is recommended that for samples containing over 2 % chlorine, the sample weight be based on the chlorine content as given in the following table:

Chlorine Content percent	Weight of Sample, g	Weight of White Oil, g
2 to 5	0.4	0.4
Over 5 to 10	0.2	0.6
Over 10 to 20	0.1	0.7
Over 20 to 50	0.05	0.7

NOTE 6—If the sample is not readily miscible with white oil, some other low sulfur combustible diluent may be substituted. However, the combined weight of sample and nonvolatile diluent shall not exceed 1.0 g or more than 0.8 g if the IP 12 bomb is used.

6.2 *Addition of Oxygen*—Place the sample cup in position and arrange the cotton wisp or nylon thread so that the end dips into the sample. Assemble the bomb and tighten the cover securely. (Caution—See Note 7.) Admit oxygen slowly (to avoid blowing the oil from the cup) until a pressure is reached as indicated in the following table:

Capacity of Bomb, ml	Minimum Gage Pressure,[a] atm kgf/cm²	Maximum Gage Pressure,[a] atm kgf/cm²
300 to 350	38 (39)	40 (41)
350 to 400	35 (36)	37 (38)
400 to 450	30 (31)	32 (33)
450 to 500	27 (28)	29 (30)

[a] The minimum pressures are specified to provide sufficient oxygen for complete combustion and the *maximum pressures represent a safety requirement.*

NOTE 7: Caution—Do not add oxygen or ignite the sample if the bomb has been jarred, dropped, or tilted.

6.3 *Combustion*—Immerse the bomb in a cold distilled-water bath. Connect the terminals to the open electrical circuit. Close the circuit to ignite the sample. (Caution—See Note 8.) Remove the bomb from the bath after immersion for at least 10 min. Release the pressure at a slow, uniform rate such that the operation requires not less than 1 min. Open the bomb and examine the contents. If traces of unburned oil or sooty deposits are found, discard the determination and thoroughly clean the bomb before again putting it in use (Note 3).

NOTE 8: Caution—Do not go near the bomb until at least 20 s after firing.

6.4 *Collection of Sulfur Solution*—Rinse the interior of the bomb, the oil cup, and the inner surface of the bomb cover with a fine jet of distilled water, and collect the washings in a 600-mL beaker having a mark to indicate 75 mL. Remove any precipitate in the bomb by means of a rubber policeman. Wash the base of the terminals until the washings are neutral to a suitable indicator. Add 10 mL of saturated bromine water to the washings in the beaker. (The volume of the washings is normally in excess of 300 mL.) Place the sample cup in a 50-mL beaker. Add 5 mL of saturated bromine water, 2 mL of HCl, and enough distilled water just to cover the cup. Heat the contents of the beaker to just below its boiling point for 3 or 4 min and add to the beaker containing the bomb washings. Wash the sample cup and the 50-mL beaker thoroughly with distilled water.

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[6] Emery Polishing Paper Grit No. 3/0 may be purchased from the Behr-Manning Co., Troy, N. Y. Chromic oxide may be purchased from J. T. Baker & Co., Phillipsburg, N. J.

APPENDIX B-12

THE ANALYSIS OF ANIONS BY ION CHROMATOGRAPHY

Quanterra Environmental Services

SOP No. STL-WC-0028
Date Initiated:11/07/96
Revision No. 0
Revision Date: N/A
Page 1 of 24

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OPERATION-SPECIFIC STANDARD OPERATING PROCEDURE

TITLE: The Analysis of Anions by Ion Chromatography

(SUPERSEDES: SL4041)

Prepared by: Margaret C. Winter

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Prepared by: _____

Reviewed by: _____
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Title: The Analysis of Anions by Ion Chromatography

1.0 SCOPE AND APPLICATIONS

1.1 This Standard Operating Procedure describes in detail the determination of fluoride, chloride, nitrite-N, bromide, nitrate-N, orthophosphate and sulfate ions in water and soil by ion chromatography.

1.2 This method is applicable to drinking water, ground and surface waters, domestic and industrial wastewater, atmospheric precipitation samples, treatment process samples and also, solid matrices.

1.3 This method is restricted to use by or under the supervision of analysts experienced in the use of ion chromatography and in the interpretation of the resulting ion chromatogram. A sample chromatogram is shown in Figure 1.

1.4 When retention times indicate possible peak misidentification, anion identification should be supported by the addition of spike solutions covering the anions of interest (i.e., the method of known additions).

1.5 Approximately 7 samples per hour can be analyzed with this method.

1.6 The detection limits for the above analytes are listed as follows:

ANALYTE	Detection Limit (mg/L)
Fluoride	0.1
Chloride	0.20
Nitrite-N	0.02
Bromide	0.25
Nitrate-N	0.02
Orthophosphate	0.50
Sulfate	0.50

NOTE: The detection limit for a specific matrix may differ from those listed above, depending upon the nature of the sample.

1.7 Responsibilities:

Analyst: to schedule and order samples requiring analysis of anions by ion chromatography, to follow this procedure and to report any abnormal results to the Wet Chemistry Group Leader.

Wet Chemistry Group Leader: (or designate) to review data prior to release from the lab.

Lab Manager: (or designate) to delegate the performance of this procedure to analysts who are qualified to perform this procedure and operate associated equipment.

2.0 SUMMARY OF METHODS

2.1 A filtered aliquot of sample is injected into a stream of carbonate-bicarbonate eluant in an ion chromatographic system consisting of a guard column, separator column, suppressor column and conductivity detector. The guard and separator columns are packed with low-capacity, strongly basic anion exchange resin. Ions are separated based on their affinity for the exchange sites of the resin. The separated anions are directed onto a suppressor column that contains cation exchange resin in the hydrogen form. The suppressor column reduces the background conductivity of the eluant to a low or negligible level (weakly conductive carbonic acid) and converts the anions to their highly conductive acid forms. The separated anions in their acid form are measured by conductivity. They are identified on the basis of retention time as compared to known standards. Quantification is accomplished by measuring the area of the resultant peaks and comparing to a calibration curve generated from known standards.

3.0 DEFINITIONS

3.1 See Quanterra Quality Assurance Management Plan (QAMP) for definitions of common laboratory terms.

3.2 Method of known additions - The single standard addition technique involving addition of a known standard to a sample to confirm the identity of a tentatively identified analyte.

3.3 Retention Time - The interval measured from the point of sample injection to the point of maximum peak height or area.

3.4 Resolution - The ability of a column to separate constituents under specified test conditions.

3.5 Eluant - The ionic mobile phase used to transport the sample through the exchange column.

3.6 Regenerant - A solution that converts and maintains an active form of the suppressor.

4.0 INTERFERENCES

4.1 Any substance that has a retention time coinciding with that of any anion to be determined will interfere. For example, relatively high concentrations of low molecular weight organic acids (e.g., acetate and formate) interfere with the determination of chloride and fluoride. A high concentration of any one ion also interferes with the resolution of others. Sample dilution overcomes many interferences. To resolve uncertainties of identification, use the method of known additions (section 5.8.5).

4.2 Spurious peaks or elevated baseline may result from contaminants in reagent water, glassware, or sample processing apparatus. Because small sample volumes are used, scrupulously avoid contamination.

4.3 Water from the sample injection will cause a negative peak or dip in the chromatogram when it elutes because its conductivity is less than that of the suppressed eluant. This dip usually occurs before fluoride and chloride. Any peak of interest eluting near the water dip must be sufficiently resolved from the dip to be accurately quantified. Adjustment of sample background may be accomplished by diluting the sample with eluant if sample dilution is required prior to analysis. Add an equivalent of 1.0 ml of a prepared eluant concentrate (solution that is 100 times more concentrated than the eluant used for analysis) per 100 ml of sample. It is important to prepare a blank using reagent water and eluant (100:1) to compensate for any anionic impurities present.

4.4 Samples and reagents containing particulates require filtration through a 0.45 mm filter to avoid fouling or clogging the resin columns.

5.0 SAFETY

5.1 Procedures shall be carried out in a manner that protects the health and safety of all Quanterra associates.

5.2 Eye protection that satisfies ANSI Z87.1 (as per the Chemical Hygiene Plan), laboratory coat, and appropriate gloves must be worn while samples, standards, solvents, and reagents are being handled. Disposable gloves that have been contaminated will be removed and discarded; other gloves will be cleaned immediately.

5.3 The health and safety hazards of many of the chemicals used in this procedure have not been fully defined. Additional health and safety information can be obtained

from the MSDS files maintained in the laboratory. The following specific hazards are known:

5.3.1 The following material is known to be **corrosive**: sulfuric acid.

5.4 Exposure to chemicals will be maintained **as low as reasonably achievable**, therefore, unless they are known to be non-hazardous, all samples will be opened, transferred and prepared in a well-ventilated area. Solvent and waste containers will be kept closed unless transfers are being made.

5.5 All work must be stopped in the event of a known or potential compromise to the health or safety of any ITAS associate. The situation must be reported immediately to a laboratory supervisor.

6.0 EQUIPMENT AND SUPPLIES

6.1 Dionex 4000i Ion Chromatograph.

6.2 Sample loop, 50 ml.

6.3 Guard column, Dionex IonPac AG4A (P/N 37042).

6.4 Anion Separator, Dionex IonPac AS4A (P/N 37041).

6.5 Membrane suppressor, Dionex Anion Micro (P/N 038019).

6.6 Conductivity detector, Dionex.

6.7 Automated sampler, Dionex.

6.8 Sample Vials, 5 ml, Dionex.

6.9 Filtercaps (For 5-mL vials), Dionex P/N 038009.

6.10 Pump, Dionex Gradient.

6.11 Interface, Dionex advanced.

6.12 Computer, IBM XT or equivalent.

6.13 AutoIon 450 Chromatography Software.

6.14 Monitor, color or monochrome.

6.15 Printer, Epson FX-850 or equivalent.

6.16 Balance, analytical.

6.17 Spatula.

6.18 Sonicator or shaker (soils only).

6.19 Eppendorf auto pipette.

6.20 Volumetric Flasks, 100-ml, 200-ml, 1-L and 2-L.

6.21 Volumetric pipettes.

6.22 Transfer pipettes.

6.23 Funnels.

6.24 Membrane filter, 0.45 m.

7.0 REAGENTS AND STANDARDS

7.1 Helium and Nitrogen, gases, ultra high purity.

7.2 Deionized or distilled water : obtained from the Millipore Unit, quality equivalent to ASTM Type II.

7.3 Sodium bicarbonate (1.0 M): Dissolve 42.0 g sodium bicarbonate ($NaHCO_3$) in reagent water and dilute to 500 ml.

7.4 Sodium carbonate (0.5M): Dissolve 26.5 g sodium carbonate (Na_2CO_3) in reagent water and dilute to 500 ml.

7,5 Eluant solution: Pipette 3.4 ml of the 1.0 M sodium bicarbonate solution (7.3) , and 7.2 ml of the 0.5 M sodium carbonate solution (7.4) in reagent water and dilute to 2 liters. The resulting solution is 1.7mM in $NaHCO_3$ and 1.8 mM in Na_2CO_3.

7.6 Regenerant solution (0.027N): Dilute 3.0 ml conc. sulfuric acid (H_2SO_4) to 4 liters with reagent water.

7.7 Stock standard solutions for Calibration and CCVS, 1000 mg/L (1 mg/ml): Stock standard solutions for each inorganic anion (Cl^-, F^-, Br^-, NO_3^--N, NO_2^--N, PO_4^{3-}, and SO_4^{2-}). Standards must be certified solutions purchased commercially. Stability of standards depends upon the manufacturer's recommendations.

7.8 Stock standard solutions for LCS, ICVS, and MS, 1000 mg/L (1 mg/ml): Stock standard solutions for each inorganic anion (Cl^-, F^-, Br^-, NO_3^-, NO_2^-, PO_4^{3-}, and SO_4^{2-}). Commercially purchased from a different source than the calibration stock standard. Stability of standards depends upon the manufacturer's recommendations.

7.9 High Intermediate Standards for Calibration and CCVS:

Ion	**ml of stock**	**mg/L**
Fluoride	1	5
Chloride	1	5
Nitrite-N	0.08	0.4
Bromide	1	5
Nitrate-N	0.20	1.0
Orthophosphate	4.0	20
Sulfate	4.0	20

Pipette the stock standard solutions (7.7) into a 200 ml volumetric flask. Dilute to volume and transfer to a 250 ml. plastic bottle. Prepare weekly. Store in the refrigerator when not in use.

7.10 Low Intermediate Standards for Calibration:

Ion	**ml of stock**	**mg/L**
Fluoride	0.4	2
Chloride	0.8	4
Nitrite-N	0.08	0.4
Bromide	1	5
Nitrate-N	0.08	0.4
Orthophosphate	2.0	10
Sulfate	2.0	10

Pipette the stock standard solutions (7.7) into a 200 ml volumetric flask. Dilute to volume and transfer to a 250 ml. plastic bottle. Prepare weekly. Store in the refrigerator when not in use.

7.11 Intermediate Standards for LCS and ICVS:

Ion	ml of stock	mg/L
Fluoride	1	5
Chloride	1	5
Nitrite-N	0.263	0.4
Bromide	1	5
Nitrate-N	0.885	1.0
Orthophosphate	4	20
Sulfate	4	20

Pipette the stock standards for LCS, ICVS, and MS (7.8) into a 1000 ml volumetric flask. Dilute to volume and transfer to a 250 ml. plastic bottle. Prepare weekly. Store in the refrigerator when not in use.

7.12 Calibration Standards:

ml of Intermediate	Concentration in mg/L						
	F^-	Cl^-	NO_2^-	Br^-	NO_3^-	PO_4^{3-}	SO_4^{2-}
5	0.10	0.20	0.02	0.25	0.02	0.5	0.5
10	0.50	0.50	0.04	0.50	0.10	2.0	2.0
20	1.0	1.0	0.08	1.0	0.20	4.0	4.0
50	2.5	2.5	0.20	2.5	0.50	10.0	10.0

Pipette 5 ml of the low intermediate standard (7.10) and 10, 20, and 50 ml of the high intermediate standard (7.9) into different 100-ml volumetric flasks. Dilute to volume with DI water. Prepare weekly.

7.13 ICVS Solution (F^-, Cl^-, Br^- = 2.0 mg/L; PO_4^{3-}, SO_4^{2-} = 8.0 mg/L; NO_3^--N = 0.40 mg/L; and NO_2^--N = 0.16 mg/L): Pipette 40 ml of the intermediate standard (7.11) into a 100 ml volumetric flask. Dilute to volume with DI water.

7.14 CCVS Solution (F^-, Cl^-, Br^- = 2.0 mg/L; PO_4^{3-}, SO_4^{2-} = 8.0 mg/L; NO_3^--N = 0.40 mg/L; and NO_2^--N = 0.16 mg/L): Pipette 40 ml of the intermediate standard (7.9) into a 100 ml volumetric flask. Dilute to volume with DI water.

7.15 Laboratory Control Sample for Waters (Fl^-, Cl^-, Br^- = 1.0 mg/L; PO_4^{3-}, SO_4^{2-} = 4.0 mg/L; NO_2^--N = 0.08 mg/L; and NO_3^--N = 0.20 mg/L): Pipette 20 ml of the intermediate standard (7.11) into 100 ml volumetric flask. Dilute to volume with DI water.

7.16 Matrix Spike for Waters (Fl^-, Br^-, PO_4^{3-} = 2.0 mg/L; Cl^-, SO_4^{2-} = 20.0 mg/L; NO_2^--N = 0.609 mg/L; NO_3^--N = 2.26 mg/L): From stock standards (7.8)

pipette 10 µl each of F^-, Br^-, PO_4^{3-}, NO_2^--N, 100 µl each of Cl^- and SO_4^{2-}, and 50 µl of NO_3^--N into a 5 ml of the appropriate samples.

NOTE: For samples to stay within the system calibration range, spiked samples must be diluted to at least 1:5 for Cl^- and 1:2 for SO_4^{2-} prior to the run.

7.17 Blank Standard for Waters: Deionized water used to prepare the standards should be used as blank standard.

7.18 Blank Standard for Soils: Solid glass beads are used as blank standard.

8.0 SAMPLE COLLECTION, PRESERVATION AND STORAGE

8.1 All samples should be collected in polyethylene or glass amber bottles.

8.2 Water and soil samples preservation and holding times for the anions are as follows:

Analyte	Preservation	Maximum Holding Time (from collection)
Fluoride	None	28 days
Chloride	None	28 days
Nitrite-N	Cool, 4 °C	48 hours
Bromide	None	28 days
Nitrate-N	Cool, 4 °C	48 hours
Orthophosphate	Cool, 4 °C	48 hours
Sulfate	Cool, 4 °C	28 days

NOTE: In all cases, samples should be analyzed as soon as possible after collection.

9.0 QUALITY CONTROL

9.1 Measure and record a method blank and laboratory control sample with every analytical batch of the same matrix containing 20 samples or less.

9.2 Run one matrix spike and duplicate sample for every 20 samples of the same matrix in each batch.

9.3 Measure and record an ICVS immediately after the last initial calibration standard.

9.4 Measure and record an ICB immediately after the ICVS.

9.5 Measure and record the CCVS after every 10 injections of samples, method blanks, or QC samples.

9.6 A CCB must be analyzed after the final CCVS following the analytical sequence.

9.7 The method blank, ICB, and CCB concentrations must always have a concentration of less than the method or contract required detection limit.

9.8 The acceptable limit for the LCS is 80 -120% or determined by control charts.

9.9 The matrix spike should be ± 25% of the true value for water samples and ± 35% of the true value for soil samples.

9.10 Duplicate sample RPD should not be greater than 20% for water and 35% for soil samples.

9.11 The correlation coefficient of calibration must be ≥ 0.995.

9.12 The CCVSs and ICVSs must be ± 15% of the true value and in the established 5% retention time windows, unless shifts in retention times can be explained by the effect of high levels of analytes in preceding runs.

9.13 Other Quality Control requirements may be Contract Specific. Refer to the appropriate QAS.

9.14 Corrective Action

9.14.1 Samples associated with method blanks or laboratory control samples which fail the criteria of this section must be prepared and analyzed again with an acceptable blank and LCS. For projects requiring matrix spike and or duplicate analysis, data will be reported with appropriate flags when the results fall outside the suggested control limits. No reanalysis will be performed for out-of-control matrix spikes or duplicates unless specifically requested by a project manager.

9.14.2 For an unacceptable ICVS or ICB, investigate instrument contamination and reanalyze the samples.

9.14.3 For an unacceptable CCVS or CCB, investigate instrument contamination and reanalyze all samples analyzed after the last acceptable CCB.

10.0 CALIBRATION AND STANDARDIZATION

10.1 The instrument is calibrated using five calibration standards in ascending order of concentration preceded by a blank standard. Reference 7.12 for calibration standards concentration, and Section 11 for calibration procedure.

10.2 An instrument calibration curve must be generated daily or once every 24 hours and each time the instrument is set up. (If the eluant is changed, a new set of calibration curves must be analyzed and documented.)

10.3 The working calibration curve must be verified after every 10 injections of samples, method blanks, and/or quality control samples. If the response for any analyte varies from the expected values by more than ± 15% or if the retention time varies by more than 5% from the expected retention time, the analyses must be repeated from the last good CCVS and CCV.

10.4 The acceptable correlation coefficient for the calibration curve is 0.995 or greater. If it is not, an acceptable calibration curve must be generated before the analysis of the sample begins.

11.0 PROCEDURE

11.1 One time procedural variations are allowed only if deemed necessary in the professional judgment of supervision to accommodate variation in sample matrix, radioactivity, chemistry, sample size, or other parameters. Any variation in procedure shall be completely documented using a Nonconformance Memo and approved by a Technical Specialist and QA Manager. If contractually required, the client shall be notified. The Nonconformance Memo shall be filed in the project file.

11.2 Any unauthorized deviations from this procedure must also be documented as a nonconformance, with a cause and a corrective action described.

11.3 Sample Preparation.

11.3.1 Water samples are analyzed neat. Use blank standard (7.17) for a method blank sample. For LCS or MS, use the standards as stated in section 7.15 or 7.16. Pipette sample into a 5-ml autosampler vial.

11.3.2 Soil samples are extracted using the instructions in section 11.3.2.1

11.3.2.1 Weigh five grams of sample (or blank solid matrix such as glass beads as stated in 7.18.) into a 50 ml centrifuge tube. If an LCS, spike the sample with 10 ml of the intermediate standards (7.11) to make Fl^-, Cl^-, Br^- = 10 µg/g; $PO4^{3-}$, SO_4^{2-} = 40 µg/g; NO_2^--N = 0.8 µg/g; and NO_3^--N = 2.0 µg/g. For MS, spike the sample with 100 µl each of Fl^-, Br^-, PO_4^{3-}, and NO_2^--N, 500 µl of NO_3^--N, and 1.0 ml each of Cl^- and SO_4^{2-} stock standards (7.8) to make Fl^-, Br^-, PO_4^{3-} = 20 µg/g; NO_2^--N = 6.09 µg/g; NO_3^--N = 22.6 µg/g; and Cl^-, SO_4^{2-} = 200 µg/g. LCS and MS must be prepared from a different source than the stock standards used for calibration. Add deionized water to the 50 ml mark. Mix the slurry for five minutes using a Vortex stirrer or sonicator device. Filter the resulting slurry before injecting using a 0.45 m membrane type filter into a 5 ml sample vial.

NOTE: In order to keep anions in the calibration range, spiked samples must be diluted to at least 1:5 for Cl^- and 1:2 for SO_4^{2-} prior to the run.

11.4 Dionex 4000i Ion Chromatograph

11.4.1 Operating Conditions

11.4.1.1 Below are the recommended operating conditions for the Dionex 4000i Ion Chromatograph used in the analysis.

CONDITIONS

Eluant	as specified in 7.5
Eluant Flow	2.0 ml/min
Regenerant	0.027N H_2SO_4
Regenerant Flow	2.5 ml/min
Column	as specified in 6.3 and 6.4
Detector	Conductivity, 10mS
Injection Volume	50 µl
Retention Time Window	5 %

11.4.2 Initial Start-Up (System Equilibration)

11.4.2.1 Before beginning operation of Dionex 4000i Ion Chromatograph make sure all of the installations are

properly connected; and the reagent (eluant, regenerant) bottles are properly filled. Load standards and samples into the autosampler as per example run summary (Table 1).

Table 1

1. Autocal 1R (blank)
2. Autocal 2R (first std 7.12)
3. Autocal 3R (second std 7.12)
4. Autocal 4R (third std 7.12)
5. Autocal 5R (fourth std 7.12)
6. Autocal 6R (7.9)
7. ICVS
8. ICB
9. Prep Blank
10. LCS
11. Samples (to a max. of 10 including Prep Blank and LCS)
12. CCVS
13. CCB

11.4.2.2 Validate order of samples and standards in the autosampler as per SOP STL-QA-0019.

11.4.2.3 Turn gases on (Helium at 20 psi and Nitrogen at 100 psi).

11.4.2.4 Turn **System** ON by pushing up system switch **Eluant Degas Module.** Also, push up position 3 for **Eluant Reservoir.**

11.4.2.5 Turn B ON for **Advanced Chromatography Module** (System 1).

11.4.2.6 Turn **Gradient Pump** to Start. Pressure on the Gradient Pump should be less than 1500 psi.

11.4.2.7 Push **Conductivity Detector** cell ON and wait approximately 15 minutes for equilibrium (i.e., when conductivity reaches between 14-16 mS).

11.4.2.8 Turn ON **Advanced Computer Interface**.

11.4.3 Programming Data Acquisition Parameters

11.4.3.1 Select the **METHOD icon** from the **AI-450 main menu.** The METHOD editor is used to create, store, or modify methods for an analysis.

11.4.3.2 A double-click on the **METHOD icon** will display the window which will have the current setting for data acquisition.

11.4.3.3 Select the **SYSTEM** for which the method is intended. In this case **ACI SYS 1: Anion/Carbohydrate System** is selected.

11.4.3.4 Select the number and kind of **DETECTOR** used for analysis. For this purpose, **DETECTOR 1 CDM-2** is selected. Also, enter **40 mS** as **Plot Scale** for detector used.

11.4.3.5 Enter the **Run Time,** in tenths of minutes (0 to 999 minutes). This is the time required for all peaks in the sample to elute. Select **8 minutes** which is usually appropriate for the ions to elute.

11.4.3.6 Select the **Sampling Rate** as **5 Hz.** samples/second to be used by the detector.

11.4.3.7 Click the **Gradient** command button to open the **Gradient Editor dialog box.**

11.4.3.8 Program gradient steps by: 1) adjusting **flow** to 2.0 ml/min, 2) **Eluant 3** as 100%, 3) **Hi limit** as 2000, 4) **Lo limit** as 700, 5) enter **Event,** 6) enter **File save** and **close** Gradient Editor dialog box.

11.4.3.9 Clicking on the **"Timed Events"** command button opens the **Timed Events Editor dialog box.**

11.4.3.10 Program Timed Event steps by editing **initial Step Time** which should have **Start, Run, Offset**, and **Autosampler** at **ON** positions. Set **Range** at 10.0 mS for **CDM-1.** Turn **B on** and set **temp.** at 25°C for **CHA.** Edit **Step Time 0.0** which must have positions **Inject** and **B** for CHA turned ON. Turn ON **Begin Sampling** for ACI and press ENTER. At **Step Time 6.2,** take **Inject off**, turn ON

autosampler, and press **ENTER.** At **Step Time 6.3** turn off **autosampler** and press **ENTER** and **save File. Close** Timed Event Editor.

11.4.4 Programming Data Processing Parameters

11.4.4.1 The **"Detector-1"** command buttons open the **Data Processing dialog** for the selected detector. The Data Processing parameters specify how you want the data from an analysis processed and the type of information you want included in your final analytical report.

11.4.4.2 The **"Integration"** command button opens the **Integration Parameters** dialog box. The Integration Parameters specify how the acquired data is processed by the computer for peak detection and integration.

11.4.4.3 Edit **Integrator Parameters Peak Width** as 5 seconds, **Peak Threshold** as 200, and **Area Reject** as 300 area counts for **Peak Detection.** For **Reference Peak, Area Reject** normally should be 300, and **Retention Time** should be set at 5.00%.

11.4.4.4 Open the **Data Events Dialog box** in which you create a list of timed-based events which affect how the raw data is detected and integrated. Enter Time (estimated retention time) for F^-. Select Void volume treatment for this peak from the Event Options list. Click on Add Event. Repeat the above procedure in same sequence for Cl^- by using Time as the estimated retention time for Cl^- This will turn on Void Volume treatment for their respective peaks. **Exit** Data Event.

11.4.4.5 Open the **Calibration Parameters** dialog box. The Calibration Parameters determine how the detected peaks are identified and quantitated.

11.4.4.6 Calibration Parameters setting are edited as **level 1** on, **Fit type** as linear, **Calibration Update** as **Replace, Standardization** as **External, Peak Calibration** as Area. Enter 1 for **sample volume, Dilution Factor, Cal Std. Volume,** and **Default Internal Std** conc in samples in the Default section of the Calibration Parameters.

11.4.4.7 Open **Component Table Dialog box.** The component Table contains the information needed to perform the calibration calculations; including the name, concentration, and retention time of each sample component.

11.4.4.8 To enter or add component information click on the **Component** button, and enter **Component Name** included on the chromatogram; enter **expected Retention Time** for the anions analyzed; specify **% Time window** as 5%; and enter 1 as **Reference Component.** At the recommended operating conditions (11.4.1), the seven common inorganic anions elute in the following order: F^-, Cl^-, NO_2^--N, Br^-, NO_3^--N, PO_4^{3-}, and SO_4^{2-}). See Figure 1 for example ion chromatogram.

11.4.4.9 Save **Data File.**

11.4.5 Programming Schedule

11.4.5.1 To start Schedule Program double-click on **SCHED icon** in the AI-450 Program Menu. The **Schedule Editor** window will appear.

11.4.5.2 Under the File pull-down menu you will find the commands "New" and "Open". The new command sets up the Schedule Table for a new Schedule. The Open command allows you to bring an existing Schedule onto the screen.

11.4.5.3 A Schedule may contain instructions for up to 99 separate injections. To enter information for a schedule step, point and click inside the desired cell.

11.4.5.4 Type in **Sample Name** (30 characters or less) to identify the sample.

11.4.5.5 Type in the **path** and **file name** of the Method you want to use to analyze the sample.

11.4.5.6 In the Data File section, type in a **file name**(seven characters or less) under which the raw data from the analysis will be stored on disk. This name takes precedence over the name specified in the Method.

11.4.5.7 Type in any dilution factor if needed in the **Dilution** section.

11.4.5.8 Save **Schedule**.

11.4.6 Starting the Run Program

11.4.6.1 To start the **Run Program** double-click on the RUN icon in the AI-450 Main Menu. After the program is loaded, a window displaying the AutoMonitor polling information appears which lists each ACI/system connected to the computer.

11.4.6.2 Select **Load Schedule** which you saved in section 11.4.5.8.

11.4.6.3 Enter **Number** of loops for the Schedule wants to be repeated. A Schedule may be repeated from 1 to 99 times. If you want the Schedule to run continuously, enter a "1".

11.4.6.4 Enter a "1" for **Starting Inject #** for the Schedule.

11.4.6.5 Start **Successive Runs** determines when the next analysis in the Schedule is started. When **Automatically** is selected, the timed events for the next Method in the Schedule are started automatically as soon as the report from the preceding analysis is completed.

11.4.6.6 When all accessories are ready for operation, turn Autosampler to **RUN**.

11.4.6.7 Issue **Start** command from the RUN menu and turn everything to Remote except autosampler. Also, turn on the chart recorder.

11.5 The analysis will proceed automatically from this point. Analyte identifications and calculations for calibrations and sample concentrations are done internally by the Dionex 4000i Ion Chromatograph computer software. Analyst judgment may be used to identify peaks that are outside the retention time windows set, but such identifications should be confirmed by the method of standard addition (11.7). Sample weight or volume, extract volume, and solid fraction are manually corrected on the data summary sheet (Figure 3).

11.6 After the initial run, if any samples are above the calibrated range, dilute and rerun analysis. Make sure that the diluted sample produce a response which is near the midpoint of full scale deflection.

11.7 If the chromatogram from the initial run fails to produce adequate resolution, or if the identification of specific anions is questionable, use the method of known additions. Add an amount of the appropriate spike standard that would yield a response approximately equal to the response of the peak being evaluated. If after the addition of the known standard, peak shape indicates the presence of an interferent, positive identification of the target analyte can not be confirmed.

11.8 Validate order of samples and standards in the autosampler after analysis is complete as per SOP No. STL-QA-0019.

11.9 The system will automatically shut down at the end of run program provided that the method for the final CCB is "**anionse**". Turn off gases and turn off System by pushing down system switch Eluant Degas Module. Also, push down position 3 for Eluant Reservoir.

12.0 DATA ANALYSIS AND CALCULATIONS

12.1 Concentration, C (liquid)

C (mg/L) = raw value × DF

where:

DF = Dilution factor

12.2 Concentration, C (solids)

If the concentration of soil is to be reported on a dry weight basis, determine the solid fraction of the sample. Refer to SOP No. CORP-WC-0002STL, Solids Determination.

$$C(mg/kg) = \frac{raw\ value \times DF \times V_{ext}}{W \times SF}$$

where:

V_{ext} = Extraction volume, in liters

W = Weight of sample extracted, in kg

SF = Solid fraction, % solids ÷ 100

12.3 Spike Recovery (%R)

For MS:

$$\%R = \frac{C_{sp} - C_s}{\text{spike added}} \times 100$$

where:

C_{sp} = Concentration in spiked sample

C_s = Concentration in unspiked sample

For LCS:

$$\%R = \frac{\text{Conc LCS found}}{\text{LCS Spike added}} \times 100$$

12.4 Relative Percent Difference for duplicate samples

$$RPD = \frac{|\text{Conc. 1} - \text{Conc. 2}|}{(\text{Conc. 1} + \text{Conc. 2}) \div 2} \times 100$$

where:

Conc. 1 = concentration found in duplicate sample 1

Conc. 2 = concentration found in duplicate sample 2

13.0 METHOD PERFORMANCE

Training Qualification:

The group/team leader has the responsibility to ensure that this procedure is performed by an analyst who has been properly trained in its use and has the required experience. Requalification must be performed annually thereafter for this procedure. The

group/team leader must document the training and performance and submit the results to the QA Manager for inclusion in associate training files.

14.0 POLLUTION PREVENTION

This procedure will be carried out in a manner consistent with all applicable federal, state and local regulations regarding pollution control and prevention. Specific controls due to the accidental release of hazardous materials can be found in the Quanterra Chemical Hygiene Plan and facility attachments.

15.0 WASTE MANAGEMENT

Waste generated in the procedure must be segregated and disposed according to the facility hazardous waste procedure. The Health and Safety Director should be contacted if additional information is required. This information shall be contained on this section.

16.0 REFERENCES

16.1 Standard Methods for the Examination of Water and Wastewater 18th Edition, Method 4110.

16.2 EPA Manual 600/4-84-017, "The Determination of Inorganic Anions in Water by Ion Chromatography", December 1989, Method 300.0.

16.3 D 4327-84, Standard Test Method for Anions in Water By Ion Chromatography, American Society for Testing and Materials, 1916 Race Street, Philadelphia, PA 19103, 1984.

16.4 Dionex 4000i Ion Chromatography Systems Manual, 1986.

16.5 Associated SOPs

16.5.1 STL-QA-0002, Standards Preparation.

16.5.2 CORP-WC-0002STL, Solids Determination.

16.5.3 STL-QA-0019, Autosampler Loading Verification.

17.0 MISCELLANEOUS

17.1 Records Management and Documentation

17.1.1 Copies of IC run schedule sheets (Example, Figure 2) are used to record sample run order and are submitted with the data for inclusion in the each project file. Data is recorded on a Data Summary Sheet (Example,Figure 3).

17.1.2 All raw data, data run logs, copies of standard logs, and quality control charts are turned over to the Document Control Coordinator after it has been reviewed and approved.

17.2 Attachments

17.2.1 Figure 1, Sample chromatogram
17.2.2 Figure 2, Run schedule sheet
17.2.3 Data Summary Sheet

Figure 1

Figure 2

Figure 3

APPENDIX C

CLP, TCL, AND TAL CONTRACT REQUIRED QUANTITATION LIMITS

1.0 VOLATILES TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS

	Volatiles	CAS Number	Quantitation Limits Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
1.	Chloromethane	74-87-3	10	10	1200	(50)
2.	Bromomethane	74-83-9	10	10	1200	(50)
3.	Vinyl Chloride	75-01-4	10	10	1200	(50)
4.	Chloroethane	75-00-3	10	10	1200	(50)
5.	Methylene Chloride	75-09-2	10	10	1200	(50)
6.	Acetone	67-64-1	10	10	1200	(50)
7.	Carbon Disulfide	75-15-0	10	10	1200	(50)
8.	1,1-Dichloroethene	75-35-4	10	10	1200	(50)
9.	1,1-Dichloroethane	75-34-3	10	10	1200	(50)
10.	1,2-Dichloroethene (total)	540-59-0	10	10	1200	(50)
11.	Chloroform	67-66-3	10	10	1200	(50)
12.	1,2-Dichloroethane	107-06-2	10	10	1200	(50)
13.	2-Butanone	78-93-3	10	10	1200	(50)
14.	1,1,1-Trichloroethane	71-55-6	10	10	1200	(50)
15.	Carbon Tetrachloride	56-23-5	10	10	1200	(50)
16.	Bromodichloromethane	75-27-4	10	10	1200	(50)
17.	1,2-Dichloropropane	78-87-5	10	10	1200	(50)
18.	cis-1,3-Dichloropropene	10061-01-5	10	10	1200	(50)
19.	Trichloroethene	79-01-6	10	10	1200	(50)
20.	Dibromochloromethane	124-48-1	10	10	1200	(50)
21.	1,1,2-Trichloroethane	79-00-5	10	10	1200	(50)
22.	Benzene	71-43-2	10	10	1200	(50)
23.	trans-1,3-Dichloropropene	10061-02-6	10	10	1200	(50)
24.	Bromoform	75-25-2	10	10	1200	(50)
25.	4-Methyl-2-pentanone	108-10-1	10	10	1200	(50)
26.	2-Hexanone	591-78-6	10	10	1200	(50)
27.	Tetrachloroethene	127-18-4	10	10	1200	(50)
28.	1,1,2,2-Tetrachloroethane	79-34-5	10	10	1200	(50)
29.	Toluene	108-88-3	10	10	1200	(50)
30.	Chlorobenzene	108-90-7	10	10	1200	(50)
31.	Ethylbenzene	100-41-4	10	10	1200	(50)
32.	Styrene	100-42-5	10	10	1200	(50)
33.	Xylenes (total)	1330-20-7	10	10	1200	(50)

	Semivolatiles	CAS Number	Quantitation Limits Water ug/L	Low Soil ug/Kg	Med. Soil ug/Kg	On Column (ng)
62.	Dimethylphthalate	131-11-3	10	330	10000	(20)
63.	Acenaphthylene	208-96-8	10	330	10000	(20)
64.	2,6-Dinitrotoluene	606-20-2	10	330	10000	(20)
65.	3-Nitroaniline	99-09-2	25	830	25000	(50)
66.	Acenaphthene	83-32-9	10	330	10000	(20)
67.	2,4-Dinitrophenol	51-28-5	25	830	25000	(50)
68.	4-Nitrophenol	100-02-7	25	830	25000	(50)
69.	Dibenzofuran	132-64-9	10	330	10000	(20)
70.	2,4-Dinitrotoluene	121-14-2	10	330	10000	(20)
71.	Diethylphthalate	84-66-2	10	330	10000	(20)
72.	4-Chlorophenyl-phenyl ether	7005-72-3	10	330	10000	(20)
73.	Fluorene	86-73-7	10	330	10000	(20)
74.	4-Nitroaniline	100-01-6	25	830	25000	(50)
75.	4,6-Dinitro-2-methylphenol	534-52-1	25	830	25000	(50)
76.	N-Nitroso-diphenylamine	86-30-6	10	330	10000	(20)
77.	4-Bromophenyl-phenylether	101-55-3	10	330	10000	(20)
78.	Hexachlorobenzene	118-74-1	10	330	10000	(20)
79.	Pentachlorophenol	87-86-5	25	830	25000	(50)
80.	Phenanthrene	85-01-8	10	330	10000	(20)
81.	Anthracene	120-12-7	10	330	10000	(20)
82.	Carbazole	86-74-8	10	330	10000	(20)
83.	Di-n-butylphthalate	84-74-2	10	330	10000	(20)
84.	Fluoranthene	206-44-0	10	330	10000	(20)
85.	Pyrene	129-00-0	10	330	10000	(20)
86.	Butylbenzylphthalate	85-68-7	10	330	10000	(20)
87.	3,3'-Dichlorobenzidine	91-94-1	10	330	10000	(20)
88.	Benzo(a)anthracene	56-55-3	10	330	10000	(20)
89.	Chrysene	218-01-9	10	330	10000	(20)
90.	bis(2-Ethylhexyl) phthalate	117-81-7	10	330	10000	(20)
91.	Di-n-octylphthalate	117-84-0	10	330	10000	(20)
92.	Benzo(b)fluoranthene	205-99-2	10	330	10000	(20)
93.	Benzo(k)fluoranthene	207-08-9	10	330	10000	(20)

3.0 PESTICIDES/AROCLORS TARGET COMPOUND LIST AND CONTRACT REQUIRED QUANTITATION LIMITS[2,3]

	Pesticides/Aroclors	CAS Number	Quantitation Limits Water ug/L	Soil ug/Kg	On Column (pg)
98.	alpha-BHC	319-84-6	0.050	1.7	5
99.	beta-BHC	319-85-7	0.050	1.7	5
100.	delta-BHC	319-86-8	0.050	1.7	5
101.	gamma-BHC (Lindane)	58-89-9	0.050	1.7	5
102.	Heptachlor	76-44-8	0.050	1.7	5
103.	Aldrin	309-00-2	0.050	1.7	5
104.	Heptachlor epoxide[4]	111024-57-3	0.050	1.7	5
105.	Endosulfan I	959-98-8	0.050	1.7	5
106.	Dieldrin	60-57-1	0.10	3.3	10
107.	4,4'-DDE	72-55-9	0.10	3.3	10
108.	Endrin	72-20-8	0.10	3.3	10
109.	Endosulfan II	33213-65-9	0.10	3.3	10
110.	4,4'-DDD	72-54-8	0.10	3.3	10
111.	Endosulfan sulfate	1031-07-8	0.10	3.3	10
112.	4,4'-DDT	50-29-3	0.10	3.3	10
113.	Methoxychlor	72-43-5	0.50	17	50
114.	Endrin ketone	53494-70-5	0.10	3.3	10
115.	Endrin aldehyde	7421-93-4	0.10	3.3	10
116.	alpha-Chlordane	5103-71-9	0.050	1.7	5
117.	gamma-Chlordane	5103-74-2	0.050	1.7	5
118.	Toxaphene	8001-35-2	5.0	170	500
119.	Aroclor-1016	12674-11-2	1.0	33	100
120.	Aroclor-1221	11104-28-2	2.0	67	200
121.	Aroclor-1232	11141-16-5	1.0	33	100
122.	Aroclor-1242	53469-21-9	1.0	33	100
123.	Aroclor-1248	12672-29-6	1.0	33	100
124.	Aroclor-1254	11097-69-1	1.0	33	100
125.	Aroclor-1260	11096-82-5	1.0	33	100

[2]There is no differentiation between the preparation of low and medium soil samples in this method for the analysis of pesticides/Aroclors.

[3]The lower reporting limit for pesticide instrument blanks shall be one-half the CRQL values for water samples.

[4]Only the exo-epoxy isomer (isomer B) of heptachlor epoxide is reported on the data reporting forms (Exhibit B).

INORGANIC TARGET ANALYTE LIST (TAL) - TABLE 1

Analyte	Contract Required Detection Limit[1,2] (ug/L)
Aluminum	200
Antimony	60
Arsenic	10
Barium	200
Beryllium	5
Cadmium	5
Calcium	5000
Chromium	10
Cobalt	50
Copper	25
Iron	100
Lead	3
Magnesium	5000
Manganese	15
Mercury	0.2
Nickel	40
Potassium	5000
Selenium	5
Silver	10
Sodium	5000
Thallium	10
Vanadium	50
Zinc	20
Cyanide	10

(1) Subject to the restrictions specified in Exhibits D and E, any analytical method specified in ILM04.0, Exhibit D may be utilized as long as the documented instrument or method detection limits meet the Contract Required Detection Limit (CRDL) requirements. Higher detection limits may only be used in the following circumstance:

If the sample concentration exceeds five times the detection limit of the instrument or method in use, the value may be reported even though the instrument or method detection limit may not equal the Contract Required Detection Limit. This is illustrated in the example below:

For lead: Method in use = ICP
Instrument Detection Limit (IDL) = 40
Sample concentration = 220
Contract Required Detection Limit (CRDL) = 3

The value of 220 may be reported even though the instrument detection limit is greater than CRDL. The instrument or method detection limit must be documented as described in Exhibits B and E.

(2) The CRDLs are the minimum levels of detection acceptable under the contract Statement of Work.

FINAL SUPPORT SAMPLING PLAN

PART II

FIELD SAMPLING PLAN

for the

ENGINEERING EVALUATION AND COST ANALYSIS
OF THE FORMER CELOTEX SITE
2800 South Sacramento Avenue
Chicago, IL 60623

Prepared for:

ALLIEDSIGNAL, INC.
MORRISTOWN, NEW JERSEY
and
THE CELOTEX CORPORATION
TAMPA, FLORIDA

MARCH 1997

Prepared by:

PARSONS ENGINEERING SCIENCE, INC.
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Parsons ES Project No. 730577

TABLE OF CONTENTS

TABLE OF CONTENTS

LIST OF FIGURES

LIST OF TABLES

TABLE OF CONTENTS

SECTION 5
FIELD ACTIVITIES - SAMPLE NETWORK RATIONALE AND DESIGN

5.1 OVERVIEW

The approach that will be taken during the field investigation of the Site and the rationale behind the sampling scheme are outlined in this section. Field sampling activities will include the investigation of three media:

- On-site soils
- Groundwater
- Sediment within the inlet of the Chicago Sanitary and Ship Canal

Various Site activities that will be performed relative to defining and documenting Site property boundaries and topographic features are discussed in Subsection 5.2.

Subsections 5.3, 5.4, and 5.5 present discussions on the rationale and sampling scheme for the investigation of soil, groundwater, and sediment, respectively. Because this sampling program is the first detailed field investigation to be performed on this Site, the sampling scheme is designed to provide the information needed to better understand (1) the types of waste present on the Site (if any), (2) the potential areas of concern on the Site (if any), and (3) the horizontal and vertical extent of the potential contaminants of concern. This sampling program has also been designed to minimize the need for a second phase of field sampling prior to the preparation of the EE/CA report, and to facilitate the collection of anticipated necessary information needed to complete the EE/CA for the Site.

Table 5.1 provides a summary of the investigative samples that will be collected from each media during this field program. The table also includes the planned analytical parameters. Field sampling procedures associated with sample collection activities are discussed in Section 6 of this FSP.

TABLE 5.1
INVESTIGATIVE SAMPLE SUMMARY

FIELD SAMPLING PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

		Analytical Parameters and Number of Investigative Samples									
		VOCs	PAHs	SVOCs	8 Metals	Cyanide	Pesticides/ PCBs	Disposal Parameters	Geotech Parameters	pH	Total Organic Carbon
Site Sector	Sample Depth										
Sec A (Soil)	0 - 6"	14	0	14	14	14	14	0	2	14	0
(14 Locations)	1 -2'	14	12	2	14	14	2	2	2	14	2
	Deep	14	12	2	14	14	2	2	2	14	2
Sec B (Soil)	0 - 6"	6	0	6	6	6	6	0	1	6	0
(6 Locations)	1 - 2'	6	5	1	6	6	1	1	1	6	1
	Deep	6	5	1	6	6	1	1	1	6	1
Sec C (Soil)	0 - 6"	3	0	3	3	3	3	0	0	3	0
(3 Locations)	1 - 2'	3	2	1	3	3	1	1	0	3	1
	Deep	3	2	1	3	3	1	1	0	3	1
Sec D (Soil)	0 - 6"	2	0	2	2	2	2	0	0	2	0
(2 Locations)	1 - 2'	2	0	2	2	2	1	1	0	2	1
	Deep	2	0	2	2	2	1	1	0	2	1
Sec E (Soil)	0 - 6"	4	0	4	4	2	4	0	1	4	0
(4 Locations)	1 - 2'	4	3	1	4	4	1	1	1	4	1
	Deep	4	3	1	4	4	1	1	1	4	1
Sec F (Soil)	0 - 6"	6	0	6	6	6	6	0	1	6	0
(6 Locations)	1 - 2'	6	5	1	6	6	1	1	1	6	1
	Deep	6	5	1	6	6	1	1	1	6	1
Sec G (Soil)	0 - 6"	5	0	5	5	5	5	0	1	5	0
(5 Locations)	1 - 2'	5	4	1	5	5	1	1	1	5	1
	Deep	5	4	1	5	5	1	1	1	5	1

TABLE 5.1
INVESTIGATIVE SAMPLE SUMMARY

FIELD SAMPLING PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

		Analytical Parameters and Number of Investigative Samples									
		VOCs	PAHs	SVOCs	8 Metals	Cyanide	Pesticides/ PCBs	Disposal Parameters	Geotech Parameters	pH	Total Organic Carbon
Site Sector	Sample Depth										
Background (Soil)	0 - 6"	1	0	1	1	1	1	0	1*	1	1
	1 - 2'	1	0	1	1	1	1	0	1*	1	1
	Deep	1	0	1	1	1	1	0	1*	1	1
Unassigned (soil) (10 locations)	0-6	5	10	0	10	10	5	5	0	10	5
	1-2	5	10	0	10	10	5	5	0	10	5
	Deep	5	10	0	10	10	5	5	0	10	5
SOILS SUBTOTAL		138	92	61	153	153	74	31	18	153	34
Sediment (in inlet)	Not Applicable	0	1	0	1	1	0	0	0	1	0
Sediment (up-gradient)	Not Applicable	0	1	0	1	1	0	0	0	1	0
Sediment (down-gradient)	Not Applicable	0	1	0	1	1	0	0	0	1	0
SEDIMENT SUBTOTAL		0	3	0	3	3	0	0	0	3	0
Groundwater (4 Site Wells)	Not Applicable	4	0	4	4	4	4	0	0	4	0

Notes

- Site sectors are shown on Figure 5.1.
- Metals analysis will be performed on both filtered and unfiltered groundwater samples collected from each well point.
- The deep soil sample will be collected at a depth of 4 to 6 feet below ground surface unless sample material collected at a deeper sample interval exhibits indications of greater soil contamination below the 4- to 6-foot interval.
- Quality control samples are described and listed in Subsection 6.5.
- Disposal parameters refer to the following analyses: TCLP VOCs, TCLP metals, reactive cyanide, reactive sulfide, flashpoint, and sulfur.
- Geotechnical parameters refer to the following analyses: porosity, permeability, bulk density, grain size, and BTU content.
- *Refers to grain size analysis only. The soil subtotal does not include these samples.

5.2 SITE CHARACTERISTICS AND BOUNDARY DELINEATION

5.2.1 General

The Site investigation will include the execution of a document and/or deed search to facilitate the assembly of the legal description for the Site. In addition, a ground control survey will be performed to (1) locate and delineate the boundaries of the property referred to as the Site, and (2) to put in place a sampling grid across the Site. An aerial survey will also be conducted to facilitate the preparation of an accurate base map for the Site. The following subsections discuss each activity in greater detail.

5.2.2 Legal Site Description

As required by the AOC, a legal description for the Site will be assembled. Existing Site information available to the Respondents will be reviewed. In addition, if deemed necessary, a deed search will be performed by culling information from existing state and county/township records, and other pertinent available public documentation (if any), and reviewing available plats of survey (current and historical). A legal survey will be performed, if necessary, to determine easements and right-of-ways relative to the Site.

5.2.3 Ground Control and Topographic Survey

A Ground Control Survey will be performed using Global Positioning System. The control system will permanently locate at least four horizontal and vertical control points within or near the boundary of the Site. The control points shall be superior or equivalent to NGS Second Order, Class II standards. Once the permanent control points are established, they will be tied into either a local site-specific system and/or the Illinois State Plane Coordinate System. Each control point will have two intervisible companion points. The ground control survey will establish a grid system across the Site at 50-foot intervals. The grid points will be used as references for documenting the location of soil borings and temporary monitoring well points.

To accurately document Site features, a topographic survey of the Site will be performed together with the ground survey. The topographic survey will document surface features such as piles/mounds, berms, foundations, on-site variations in ground elevations, etc. The scale of the map will be at least 1" = 100', and the contours will have at least 2-foot intervals.

5.2.4 Aerial Survey

Aerial photography and digital mapping of the Site (approximately 24 acres) and the surrounding area within a one-mile radius of the Site will be accomplished by stereo-digitizing directly onto a computer graphic system, at a scale of 1" = 100'. Planimetric features that are readily visible and identifiable from aerial photography will be recorded in digital form on a topographic map, including contours at 2-foot intervals. A base map of the Site (with a scale of 1" = 100' and 2-foot contours) will be prepared from the topographic map. As specified in the Scope of Work, the base map will illustrate the locations of wetland areas, flood plains, water features, drainage patterns, tanks, buildings, utilities, paved areas, and other pertinent features that may be present within the surveyed area. Due to the potential for heavy vegetation, the aerial survey will be conducted during a period of minimum vegetation and minimum snow cover, such as early spring.

5.3 SOIL INVESTIGATION

5.3.1 Sample Location Rationale

The soil sampling program has been designed to provide information on potential areas of contamination that may exist on the Site. It is known that the Site formerly housed several storage tanks containing asphalt manufacturing or petroleum products. Other areas of the Site are known to have been overlain by buildings used for either manufacturing operations or warehousing.

To simplify the Site investigation process, the Site has been subdivided into seven sectors, numbered A through G (Figure 5.1). The Site sectors were delineated based on

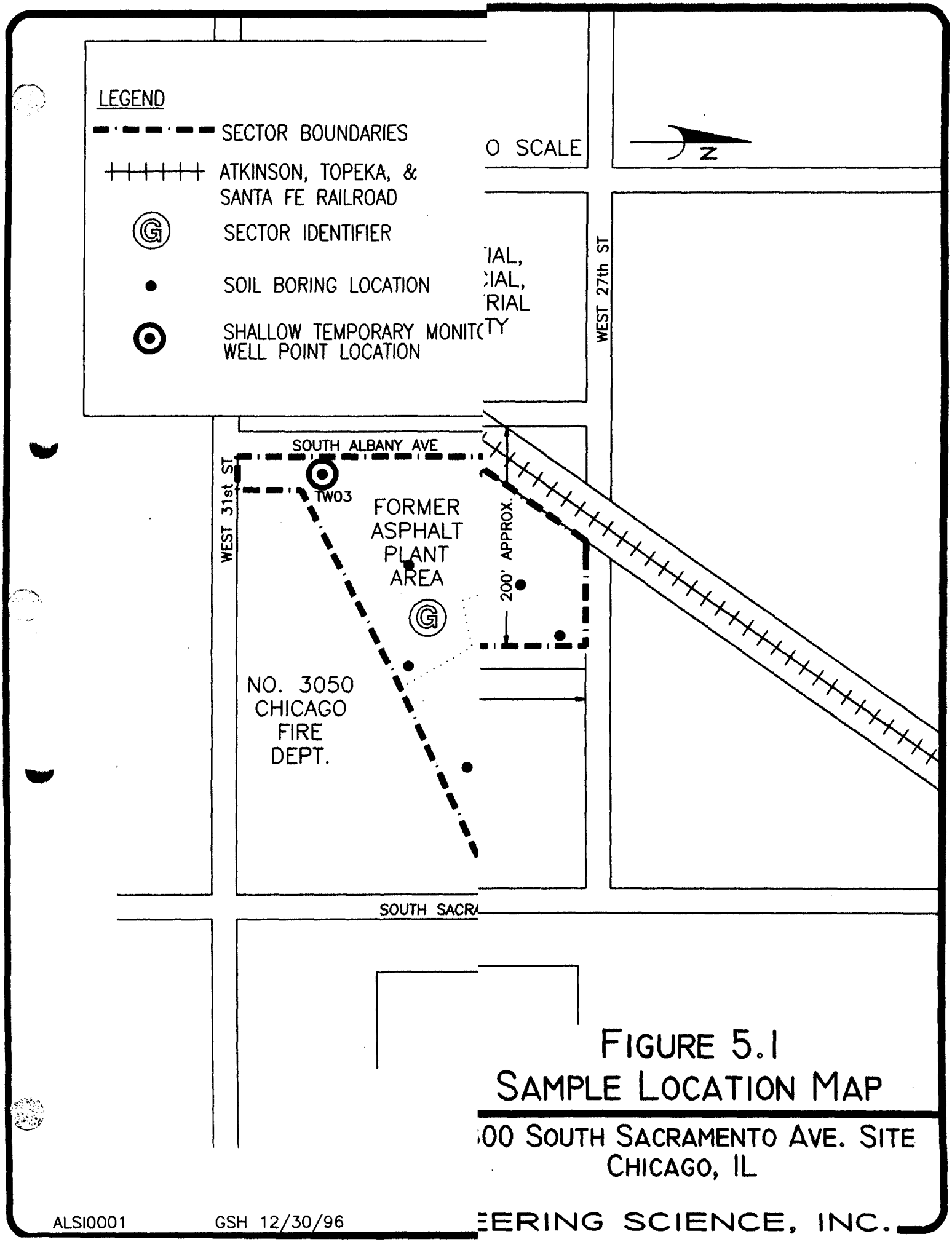

FIGURE 5.1
SAMPLE LOCATION MAP

00 SOUTH SACRAMENTO AVE. SITE
CHICAGO, IL

ALSI0001 GSH 12/30/96

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existing Site boundary features and based on information related to former Site activities. The soil sampling locations are situated within each sector and will be tied into a grid system. This grid system will be established by licensed land surveyors prior to the commencement of the field sampling program. The grid across the Site will be based on 50-foot by 50-foot spacing.

Sample locations were assigned for a variety of reasons. In each sector where practical, some sample locations have been situated close to the property boundaries to assess whether contamination (if any) extends toward these Site limits. Sample locations also are situated in the vicinity of former storage tanks and known manufacturing buildings to assess whether and to what extent operations associated with these areas have impacted the surroundings.

The approximate boundaries that delineate the various sectors and the placement of the soil boring locations are shown on Figure 5.1. Sector A has the most sample locations because it is known to have formerly housed the tank farm and is thought to have the greatest potential for "hot spots" of contamination. Sectors B and C have relatively fewer sample locations. Both sectors are partially covered by concrete and Sector C was reportedly a former parking lot. However, Sector B may have housed a storage warehouse and a few storage tanks at one time; therefore, the chosen sample locations will provide information on whether a contaminant problem exists in these areas. Sample locations in Sector D will provide data on area contamination (if any) related primarily to former storage tanks.

The sample locations in Sectors E and F are placed in areas within and on the outside of the former manufacturing building area. The data from these sample locations will enable a determination to be made as to whether the operations associated with the manufacturing area potentially impacted locations beneath building structure and/or areas

immediately surrounding the building. The sample locations in Sector G are intended to assess whether any impact from the former asphalt plant currently exists in this area.

In addition to the on-site soil boring locations, one off-site soil boring will be advanced in the Douglas Park area for background comparison of investigative samples, i.e., comparison to naturally occurring/area background levels of the target analytes. Douglas Park is situated approximately three-quarters of a mile north of the Site. This location was chosen based upon information and analytical data from the Illinois Environmental Protection Agency (IEPA). The IEPA information states that the soils in the Douglas Park area appear to be relatively undisturbed. It is therefore assumed that this area has not been affected by anthropogenic activities. Alternate and/or additional background locations may be evaluated and sampled if field observations (such as soil classification) suggest that Douglas Park is unacceptable as a background location.

In addition to the soil sample locations previously discussed, up to 10 miscellaneous (currently unassigned) locations may be added during the execution of the field sampling program. These locations will be chosen based on field observations during sampling activities. On-site characteristics such as obvious soil erosional gullies, denuded site areas, stained or discolored soil, and other noteworthy aberrant area features could prompt further soil boring investigations. The unassigned soil borings may also be used to supplement the investigation of a sector, if deemed appropriate during the execution of the field program.

5.3.2 Soil Sample Collection Approach

At each designated soil sample location, a soil boring will be advanced until either the groundwater table is encountered or a 20-foot depth below ground surface is reached, whichever occurs first. The vertical extent of contamination will be evaluated by the collection of selected soil samples along each soil boring sample core.

Soil samples for laboratory analysis will be collected from two or three sample depths at designated locations. Based on the fact that the cover soils across most of the Site

originated from an off-site location, surface soil samples will be collected and analyzed to ascertain the levels of hazardous substances (if any) present in the material. The evaluation of surface soils will also provide insight into whether surface water runoff has had an adverse impact on surface soils. For purposes of this sampling program, surface soil sampling will occur from the 0- to 6-inches depth.

To assess whether contamination is present in the shallow subsurface soils, soil samples will be collected from the 1- to 2-foot interval. A deep soil sample will be collected from each soil boring at the sample interval below the 2-foot depth that exhibits the greatest contaminant indicators. These indicators will include visual and olfactory observations, and elevated head space readings (based upon soil screening with a photoionization detector [PID]). If none of the contaminant indicators provide guidance on the choice of sample depth, the deep sample will be collected from the 4- to 6-foot interval. As discussed in Section 2.2 of the QAPP, current information on the depth to groundwater suggests that the water table could vary between 6 and 10 feet below ground surface in undisturbed areas. The default sampling depth of 4 to 6 feet below ground surface was chosen as the most probable sampling interval that would be above the saturated zone.

It is possible that coal-tar–based wastes may be encountered during the execution of this soil sampling program. If an entire soil boring exhibits an appearance of coal tar, only one subsurface sample will be collected from the boring. The sample will be taken from the deepest sample interval above the water table that exhibits the material. If several soil borings exhibit a coal-tar–based substance, field judgment will be used to determine if all, some, or none of the additional borings will be sampled. Contaminant indicators such as visual and olfactory observations, and PID readings will be used as a guide. For example, if one coal-tar mixture has elevated volatile organic compound (VOC) headspace readings and another has a green, oily, slime coating and no VOCs, this suggests that these coal-tar substances may have different chemical compositions. In instances when the contaminant indicators suggest that the constituents within various coal tar deposits on the Site may vary

from area to area, several samples of coal-tar materials will be collected. Otherwise, the location of the coal-tar material and the depth at which it was discovered will be documented in the field log book, but a sample will not be taken from the boring.

5.3.3 Analytical Approach

5.3.3.1 Overview

Based in part on existing analytical data and on information on the former Site operations, it has been determined that all soil samples will be analyzed for polynuclear aromatic hydrocarbons (PAHs), eight metals (arsenic, barium, cadmium, chromium, mercury, selenium, silver, and lead), cyanide, and pH, to characterize subsurface Site contamination. Some of the PAH data will be generated from semivolatile analyses.

Given the former presence of several storage tanks and the use of petroleum products at the Site, and the documented presence of elevated VOCs in soil samples collected from some areas of the Site that formerly housed storage tanks, all soil samples in each sector will be analyzed for VOCs. In addition, some soil samples will be analyzed for semivolatile organic compounds (SVOCs). The samples on which SVOC analyses are performed will not be analyzed separately for PAHs since the PAH list of compounds is a subset of SVOCs, and the SVOC analyses will provide PAH data.

To facilitate a preliminary assessment of potential soil management and remedial action alternatives considerations, a limited number of soil samples will be analyzed for (1) disposal parameters (reactive cyanide, reactive sulfide, flash point, sulfur, the eight metals via toxicity characteristic leaching procedure [TCLP], and TCLP VOCs); (2) pesticides/polychlorinated biphenyls (PCBs); (3) total organic carbon (TOC); and (4) geotechnical parameters (porosity, permeability, bulk density, grain size, and BTU content).

5.3.3.2 Surface Soils Analytical Parameters

Surface soil samples will be collected from all sampling locations in all sectors (Sectors A through G). All surface soil samples will be analyzed for VOCs, SVOCs, the eight total metals, cyanide, pesticide/PCBs, and pH. Ten percent of the surface soil samples in each sector (at least one sample) will also be analyzed for the geotechnical parameters.

5.3.3.3 Shallow and Deep Subsurface Soils Analytical Parameters

All samples collected from within each sector at the 1- to 2-foot depth and the deeper sample interval will be analyzed for VOCs, PAHs, the eight metals, cyanide, and pH. Approximately 10 percent (at least one sample) will be analyzed for SVOCs, pesticide/PCBs, disposal parameters, geotechnical parameters, and TOC. The sample(s) that exhibit the greatest indication of contamination will be chosen for these analyses. As previously mentioned, the contaminant indicators will include visual and olfactory observations, and headspace PID readings.

It is assumed that any miscellaneous (currently unassigned) soil boring samples collected during the soil sampling program will only be analyzed for the main contaminants of interest (PAHs, the eight metals, cyanide, and pH). If headspace screening indicates elevated readings, some of the sample(s) exhibiting these characteristics will also be analyzed for VOCs. If these samples exhibit characteristics unlike any others noted elsewhere on the Site, some of these samples will be analyzed for all parameters (VOCs, PAHs, pesticides/PCBs, the eight metals, cyanide, disposal parameters, pH, and TOC).

5.4 GROUNDWATER INVESTIGATION

Current hydrogeologic information on groundwater in the vicinity of the Site indicates that groundwater flows to the southeast, i.e., toward the Chicago Sanitary and Ship Canal. As indicated in the QAPP, the Site is located in a mixed use area that is heavily

commercial/industrial, in addition to being residential in nature. Groundwater quality within the section of Chicago in which the Site is located is not currently considered acceptable for human consumption, and the entire Chicago area receives its potable water from Lake Michigan. Furthermore, as discussed and agreed upon between the Respondents and the USEPA Region V, the sampling program associated with Site groundwater is designed to provide a general macroscopic overview (a screening assessment) of the groundwater quality relative to the Site. As such, the groundwater investigation presented herein is not intended as a detailed investigation.

Temporary groundwater monitoring well points will be advanced at four sample locations investigated during the soil boring program. Temporary well point TW01 will be situated in the western corner of the Site and is considered the upgradient location. Temporary well points TW02 and TW03 will be situated in the northern and southern sections of the Site, respectively. These well points will provide information on the water quality in these areas and information on the groundwater flow direction at the Site. Temporary well point TW04 will be located in the southeast corner of the Site. It is situated in the reported downgradient position and will facilitate the evaluation of groundwater quality prior to its departure off-site.

All groundwater samples will be analyzed for VOCs, SVOCs, pesticides/PCBs, the eight total metals (via the analysis of both filtered and unfiltered field samples), and cyanide. In addition, all groundwater samples will be field screened to determine groundwater specific conductance, temperature, and pH.

The temporary well points will be removed and abandoned in accordance with procedures described in Section 6.3.3, after the water sample is collected.

5.5 SEDIMENT INVESTIGATION

In accordance with the requirements of the AOC, the sediments within the inlet of the Chicago Sanitary and Ship Canal will be sampled. As discussed and agreed upon between

the Respondents and the USEPA Region V, the sampling program associated with the sediments is not intended to be a detailed investigation but a general screening measure for information purposes only.

One sediment sample will be collected from within the inlet at the closest accessible location that is near the Sacramento Avenue and 31st Street intersection. In addition, if conditions (such as accessibility and weather) allow, one sediment sample will be collected from within the canal itself (not within the inlet), upgradient and downgradient of the location where the inlet connects with the main body of the canal. Approximate sediment sample locations are shown on Figure 5.2. The data from these additional samples will be used for comparison purposes. All sediment samples will be analyzed for PAHs, the eight metals, cyanide, and pH. The physical characteristics of the sediment samples (odor, color, and lithology) will be evaluated in the field by the Parsons ES geologist based on olfactory and visual observations.

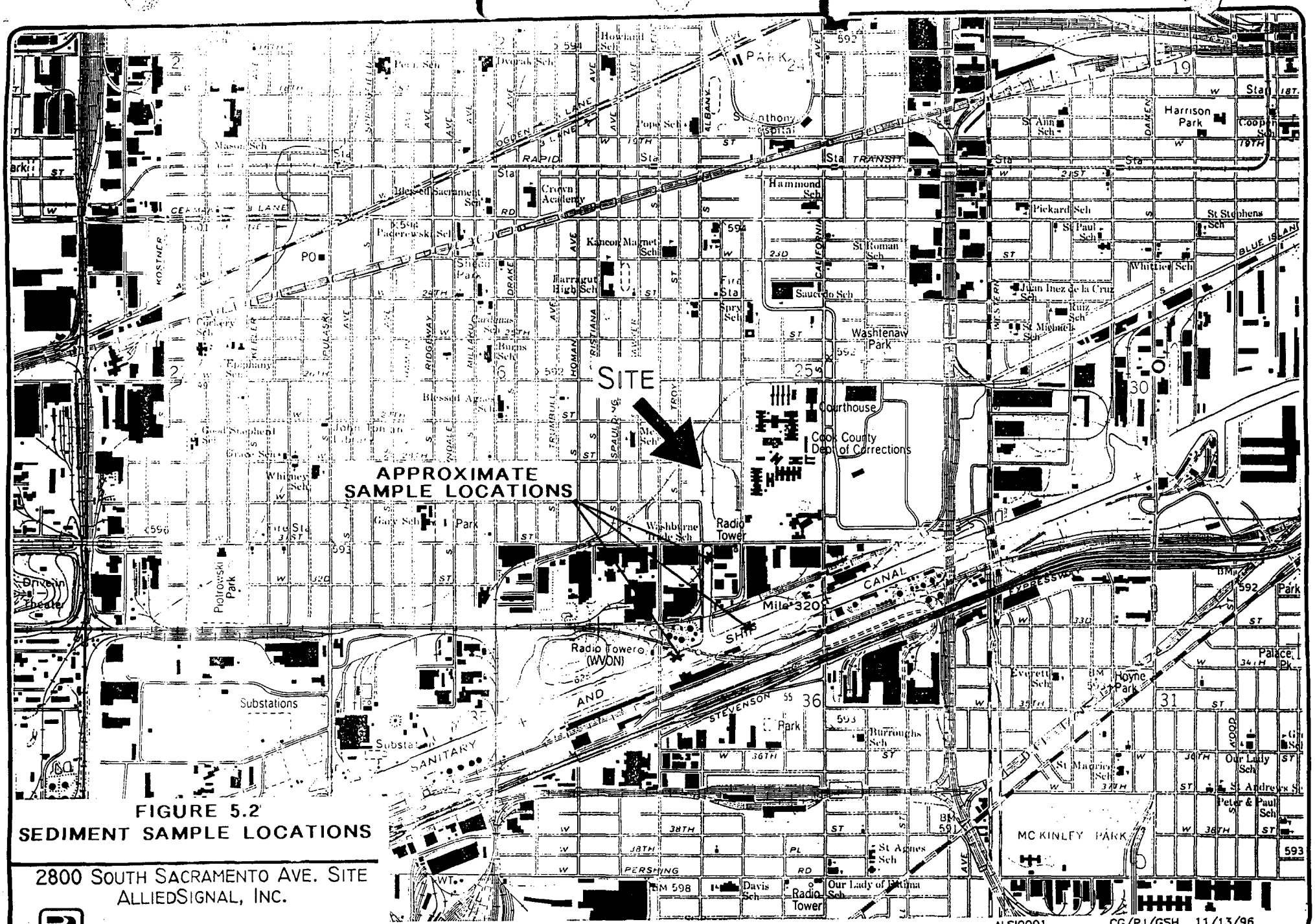

FIGURE 5.2
SEDIMENT SAMPLE LOCATIONS

2800 SOUTH SACRAMENTO AVE. SITE
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ALSI0001 CG/PJ/GSH 11/13/96

material. After sample homogenization is completed, sample jars for SVOCs/PAHs, the eight metals, cyanide, pesticide/PCBs, disposal parameters, pH, and TOC will be filled.

After this task is completed, the soil headspace in the Ziploc® bags will be screened with a PID/FID, in accordance with the protocols outlined in Subsection 6.7 and the results documented.

The split-spoon sampler from the 2- to 4-foot interval will then be retrieved. The soil core will be screened and lithology and headspace documented, but soil samples will not be containerized. The split-spoon sample from the 4- to 6-foot interval will be handled as previously discussed for the 1- to 2-foot sample, including the containerization of soil samples.

If groundwater is not encountered and sampling proceeds beyond the 6-foot depth, each subsequent 2-foot soil core will be screened, its lithology and headspace documented, and its contaminant indicators assessed and documented. If these indicators suggest that contamination is greater at a depth other than the 4- to 6-foot depth, soil from this deeper interval will be containerized as discussed previously for all the parameters and the 4- to 6-foot soil sample will be discarded. If the contamination does not appear to be greater at the deeper interval, a sample will not be collected at the new depth. The sample screening and contaminant indicator comparison and evaluation process will continue at each subsequent sample depth until either groundwater or a 20-foot depth is reached. At the end of each soil boring no more than three investigative soil samples will be retained from that boring for potential laboratory analysis.

When all soil boring samples from within a sector have been collected, the PID headspace readings and other visual/olfactory observations will be reviewed to determine which of the subsurface (below the 6-inch interval) soil samples retained for SVOC, pesticide/PCB, disposal parameter, and TOC analyses will actually be sent to the laboratory for analysis. The sample(s) exhibiting the greatest degree of contamination will be chosen.

stainless-steel bowl. The water will be decanted. At each sample location, the dredge will be lowered as many times as is necessary to collect sufficient sample material to fill all required sample jars. The sample will then be homogenized in accordance with Subsection 6.6, and the required sample containers will then be filled.

The collection of sediment samples from the canal, by hand, from areas adjacent to the canal bank is considered unsafe because the composition and integrity of the canal base is unsuitable for wading. Therefore, if sediment sampling in the canal using a boat is not possible and/or practicable due weather or other conditions, no sediment samples will be collected from the canal during this sampling program.

6.5 FIELD QUALITY CONTROL SAMPLE COLLECTION PROCEDURES

6.5.1 Field Duplicate Samples

For this field program, field duplicate samples will be collected at a frequency of approximately one duplicate sample for every 10 investigative soil, groundwater, or sediment samples. A summary of the estimated number of field duplicate samples that will be collected and analyzed for this field program is presented in Table 6.1.

Each duplicate sample will be collected from the same sample location and analyzed for the same parameters as the investigative sample. When collecting the VOC aliquot of the field duplicate sample, the sample containers for the duplicate sample aliquot will be filled directly after the investigative VOC sample containers. The remaining sample containers for the field duplicate sample will also be filled in the same alternating fashion with the investigative sample aliquots, as described for the VOC aliquots.

When sample homogenization is required, only one homogenizing process will be performed for the investigative and duplicate sample combined. Therefore, sufficient sample material necessary for filling all investigative and replicate sample containers will be collected during sampling. The duplicate sample will be submitted "blind" to the laboratory.

TABLE 6.1
SUMMARY OF INVESTIGATIVE AND QUALITY CONTROL SAMPLES

FIELD SAMPLING PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Matrix	Analyses	Estimated Total No. of Investigative Samples	Estimated No. of Field Duplicate Samples	Estimated No. of Field Rinseate Blank Samples	Estimated No. of MS/MSD Samples[1]	Matrix Total[2]
Soil						
	VOCs	138	14	0	7	152
	PAHs	92	10	0	5	102
	SVOCs	61	7	0	4	68
	8 Metals[3]	153	16	0	8	169
	Cyanide	153	16	0	8	169
	Pesticides/PCBs	74	8	0	4	82
	Disposal Parameters[4]	31	0	0	0	31
	Geotechnical Parameters[5]	21	0	0	0	21
	pH	153	0	0	0	153
	TOC	38	0	0	0	38
Sediment						
	VOCs	0	0	0	0	0
	PAHs	3	1	0	1	4
	SVOCs	0	0	0	0	0
	8 Metals[3]	3	1	0	1	4
	Cyanide	3	1	0	1	4
	Pesticides/PCBs	0	0	0	0	0
	Disposal Parameters[4]	0	0	0	0	0
	Physical Parameters[6]	3	1	0	0	4
	pH	3	1	0	1	4
	TOC	0	0	0	0	0

TABLE 6.1
SUMMARY OF INVESTIGATIVE AND QUALITY CONTROL SAMPLES

FIELD SAMPLING PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Matrix	Analyses	Estimated Total No. of Investigative Samples	Estimated No. of Field Duplicate Samples	Estimated No. of Field Rinseate Blank Samples	Estimated No. of MS/MSD Samples[1]	Matrix Total[2]
Groundwater						
	VOCs	4	1	1	1	6
	PAHs	0	0	0	0	0
	SVOCs	4	1	1	1	6
	8 Metals[3] (filtered)	4	1	1	1	6
	8 Metals[3] (unfiltered)	4	1	1	1	6
	Cyanide	4	1	1	1	6
	Pesticides/PCBs	4	1	1	1	6

Notes:

(1) Matrix spike/matrix spike duplicate (MS/MSD) samples are not additional samples, but investigative samples on which MS/MSD analyses are performed. This column also includes spike/duplicate or MS/MSD samples for inorganic analyses.

(2) The matrix total does not reflect MS/MSDs as additional samples. Trip blank samples are not included in the matrix total. One trip blank will be included with each shipment of VOC groundwater samples. Trip blanks will only be analyzed for VOCs.

(3) The 8 metals are arsenic, barium, cadmium, chromium, mercury, selenium, silver, and lead.

(4) Disposal parameters refer to TCLP 8 metals, TCLP VOCs, reactive cyanide, reactive sulfide, flash point, and sulfur.

(5) Geotechnical parameters refers to porosity, permeability, bulk density, grain size, and BTU content. * Three of these soil samples will be analyzed for grain size only.

(6) Physical parameters refers to odor, color, and lithology. These parameters will be determined in the field by the field geologist via olfactory and visual observations.

6.5.2 Matrix Spike/Matrix Spike Duplicate Samples

Matrix spike/matrix spike duplicate (MS/MSD) samples are investigative samples on which MS/MSD analysis are performed. For this field sampling program, MS/MSD samples will be collected at a frequency of one MS/MSD sample for every 20 investigative samples per matrix (soil, groundwater, and sediment). A summary of the estimated number of MS/MSD samples that will be collected and analyzed for this field program is presented in Table 6.1

To facilitate the extra analyses that will be performed by the laboratory, extra sample volume will be required by the laboratory. Therefore, for all sample matrices and media, the sample(s) identified for MS/MSD analysis will have either double or triple the typically required sample volume collected by the Parsons ES sampling team.

Because the MS/MSD sample is an investigative sample, all sample analytical requirements and sample collection and handling requirements discussed for investigative samples will also apply to the MS/MSD sample. The MS/MSD sample(s) will be clearly identified on the chain-of-custody (COC) record.

6.5.3 Field Rinseate Blanks

Field rinseate blanks will be collected at a frequency of one rinseate blank sample for every 10 or less investigative groundwater samples collected during the field program. The rinseate blanks will be created by pouring analyte free (deionized) water over and through the sampling equipment after it has been decontaminated and before it has been used to collect the next sample. A summary of the estimated number of field rinseate blank samples that will be collected and analyzed during this field program is presented in Table 6.1

The groundwater rinseate samples will be collected using the identical sampling mechanism used to collect the investigative samples. For this sampling program, the

groundwater sample equipment is a Teflon® bailer. The analyte-free deionized water will be poured over and through a decontaminated Teflon® bailer, and the rinseate liquid will be collected in the individual sample containers. The metals aliquot of the rinseate blank also will be field filtered similar to an investigative sample using an unused, sterilized, 0.45-micron filter disposable unit. If the peristaltic pump is used, the deionized water will be circulated through the pump in a manner identical to that used to collect the investigative groundwater sample. An new, unused length of sample tubing will be used to run the rinseate sample through the pump, similar to the process used for the investigative sample. At no time will the VOC sample aliquot be filtered.

Rinseate blank samples will be analyzed in the same manner and for the same range of parameters as the investigative samples. As such, rinseate samples will be analyzed for the groundwater sample parameters VOCs, SVOCs, pesticides/PCBs, the eight metals (filtered and unfiltered), cyanide, and pH, as shown on Table 6.1. All sample documentation, custody, and handling protocols used for groundwater investigative sample will also apply to the rinseate blank sample.

6.5.4 Trip Blank Samples

A trip blank sample will be enclosed in and will accompany every shipment of VOC groundwater samples sent for laboratory analysis. The trip blank sample will originate at the laboratory. It will travel with the VOC groundwater sample containers from the laboratory to the Site, accompany the VOC groundwater sample containers to the field sampling location, and will return with the filled VOC sample containers to the laboratory for analysis. The trip blank sample will consist of one VOC sample vial filled with organic-free water. At no time will the trip blank sample vial be opened at the Site or by anyone other than the laboratory.

samples to be used for MS/MSD analysis. For all media MS/MSD samples, the letters "MMSD" will be added to the end of the investigative sample series code. Since the investigative sample identified for MS/MSD analyses will be collected at double or triple the normal sample volume, all sample containers will be identified with the same sample name and contain the letters MMSD after the series code. As indicated previously, the MS/MSD sample will be identified as such on the COC form . An example of a MS/MSD sample identifier is shown below.

- SSAS1-SCA-SB01-SS01-0/6MMSD: South Sacramento Avenue Site phase 1 field investigation, site Sector A, soil boring location one, first soil sample collected from 0- to 6-inches depth. The sample will be analyzed as a MS/MSD sample by the laboratory.

7.2 FIELD CHAIN-OF-CUSTODY PROCEDURES

Field COC procedures begin as soon as a sample is collected. The sampler, now sample custodian, will label the sample containers. The sample container label (Figure 7.1) will contain sufficient information to identify the sample and the requested laboratory analyses. All sample container labels will be completed with indelible ink. The label will be affixed to the bottle and then secured in place with clear tape.

The information on the label will include, but will not be limited to:

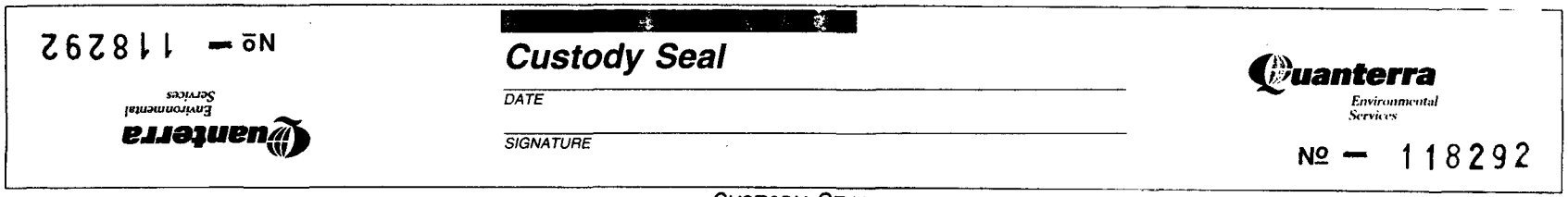
- Project name (2800 South Sacramento Avenue Site)
- Sample name, e.g., SSAS1-SCA-SB01-SS01-0/6
- Collection date, e.g., 3/29/97
- 24-hour formatted collection time, e.g., 1500 hours instead of 3:00 P.M.
- Sampler(s) name or initials, e.g., JQP
- Requested analyses with method number, e.g., VOC, SVOCs, etc.
- Preservative, if required, e.g., hydrochloric acid (HCL)

Each investigative and QC sample collected during the field investigation will be documented on a Parsons ES COC record (Figure 7.2). The COC record will be completed for all samples submitted for laboratory analysis. The Parsons ES field team

Quanterra

Client:_____ Date:_____
Site:_____ Time:_____
Sample ID:_____
Analysis:_____
Signature:_____

SAMPLE CONTAINER LABEL



CUSTODY SEAL

FIGURE 7.1
EXAMPLE OF THE SAMPLE
CONTAINER LABEL & CUSTODY SEAL

2800 SOUTH SACRAMENTO AVE. SITE
CHICAGO, IL

PARSONS ENGINEERING SCIENCE, INC.

FIGURE 7.2

1000 Jorie Boulevard, Suite 250, Oak Brook, Illinois 60521-2233
Voice 708/990-7200, Fax 708/990-7218

Chain-of-Custody Record

COC No. 000230

PROJECT NUMBER	PROJECT NAME/LOCATION	PRESERVATIVES (All samples cooled to 4°C)

PROJECT MANAGER:

ANALYSES REQUESTED

SAMPLER(S) NAME: (Please Print)

(Signature)

Date MM/DD/YY	Military Time	Comp	Grab	Sample Identification	Matrix	Container	REMARKS

Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time	Airbill #:
				Cooler #:
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time	Laboratory:
				Attn.:
Relinquished by: (Signature)	Date / Time	Received by: (Signature)	Date / Time	Send Report to:
				TAT Expected:

DISTRIBUTION: Pink Sampling Coordinator - White and Yellow Accompany Shipment - White Returned with Report

SECTION 8
SAMPLE PRESERVATION, PACKING, AND SHIPPING

8.1 SAMPLE PRESERVATION REQUIREMENTS

The sample containers, preservation requirements and holding times for Site samples are presented in Table 8.1 for water and Table 8.2 for soil/sediment.

8.2 SAMPLE PACKING AND SHIPPING

The samples collected during the Site field investigation will be handled to protect the samples from damage and to ensure sample delivery to the laboratory in sufficient time to preserve the analytical holding times. The empty sample containers will be kept in a cooler with ice to pre-chill the containers prior to sample collection. The sample bottles will be wrapped in protective covering (bubble wrap material) and enclosed in leakproof sealable bags (Ziploc® bags or equivalent).

The cooler will contain additional packaging, as necessary, to protect the sample bottles from breakage. The cooler also will contain ice to maintain the samples at approximately 4° Celsius. The ice will be encased inside two leakproof sealable bags to minimize water leakage. The laboratory copies of the COC record will be placed inside a plastic bag and affixed to the lid of the cooler.

The cooler then will be sealed for shipment using some form of shipping tape or duct tape. Custody seals will be affixed to the outside of the cooler to maintain custody of the samples during shipment. Two custody seals will affixed to the shipping cooler. The seals will be placed in the front and back of the cooler lid and will be situated across the gap between the container base and the lid so that the custody seals will break if the shipment container is opened. The custody seals will be uniquely numbered and will be covered with waterproof clear tape to prevent accidental damage during shipment. The custody seal numbers will be documented on the COC record enclosed in the cooler.

TABLE 8.1
SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
GROUNDWATER MATRIX

FIELD SAMPLING PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Parameter	Sample Container[1]	Preservative[2]	Holding Time[3]
VOCs	3 - 40 mL glass vials with septum cap	0.5 mL 36% HCL (pH<2)	14 days
SVOCs/PAHs	2 - 1L amber glass bottles with teflon liner	Cool 4+/- 2°C	7 days to extract 40 days to analyze after extraction
Pesticides/PCBs	2 - 1L amber glass bottles with teflon liners	Cool 4 +/- 2°C	7 days to extract 40 days to analyze after extraction
Cyanide	1 - 250 mL HDPE bottle	2 mL 10 N NaOH (pH>12)	14 days
7 Metals (excluding mercury)[4]	1 - 1L HDPE bottle	5 mL 35% HNO_3 (pH<2)	180 days
Mercury[4]	1 1L HDPE bottle	5 mL 35% HNO_3 (pH<2)	28 days

Notes:

1 Triple volume will be required for MS/MSD (organics) and double volume will be required for MS/duplicates (inorganics).

2 All samples will be maintained at 4 +/- 2°C upon receipt and prior to disposal.

3 Holding Times are from the date of sample collection

4 The filtered and unfiltered metals aliquots will each have a separate 1L HDPE sample container.

HDPE - High Density Polyethylene
HCL - Hydrochloric Acid
NaOH - Sodium Hydroxide
HNO_3 - Nitric Acid

TABLE 8.2
SAMPLE CONTAINERS, PRESERVATIVES, AND HOLDING TIMES
SOIL/SEDIMENT MATRIX

FIELD SAMPLING PLAN
2800 SOUTH SACRAMENTO AVENUE SITE
CHICAGO, ILLINOIS

Parameter	Sample Container[1]	Preservative[2]	Holding Time[3]
VOCs	1 - 120 mL wide mouth glass jar	Cool 4 +/- 2°C	14 days
SVOCs/PAHs	1 - 4 oz. wide mouth glass jar	Cool 4 +/- 2°C	14 days to extract 40 days to analyze after extraction
pH	Sample material will be taken from SVOC/PAH 1 - 4 oz. wide mouth container	Cool 4 +/- 2°C	14 days to leach 1 day from leach to analysis
Pesticides/PCBs	1 - 4 oz wide mouth glass jar	Cool 4 +/- 2°C	14 days to extract 40 days to analyze after extraction
8 Metals/Cyanide	1 - 8 oz. glass jar	Cool 4 +/- 2°C	180 days (except Hg and Cyanide) 28 days (Hg) 14 days (Cyanide)
TCLP VOCs	1 - 4 oz. wide mouth glass jar	Cool 4 +/- 2°C	14 days to TCLP extraction, 14 days from TCLP extraction to analysis
TOC	1 - 16 oz. wide mouth glass jar *	Cool 4 +/- 2°C	28 days
TCLP 8 Metals	* Sample material taken from the same jar as TOC	Cool 4 +/- 2°C	180 days to TCLP extraction, 180 days from TCLP extraction to analysis, except Hg, which is 28 days
Reactive cyanide and sulfide	* Sample material taken from the same jar as TOC	Cool 4 +/- 2°C	Not specified
Flash Point (closed cup)	* Sample material taken from the same jar as TOC	None required	Not specified
Sulfur	1 - 4 oz. wide mouth glass jar *	Cool 4 +/- 2°C	28 days
BTU Content[4]	Sample material taken from the same jar as sulfur	Cool 4 +/- 2°C	Not specified

Notes:

1 No additional sample volume will be required for MS/MSD or MS/duplicates analyses.

2 All samples are maintained at 4 +/- 2°C upon receipt and prior to disposal.

3 Holding Times are from the date of sample collection.

* All identified parameter analyses will be performed on sample material collected in 1 - 16 oz wide mouth glass jar.

4 The remaining geotechnical parameter samples will be collected in one galvanized steel Shelby Tube.

All investigative samples will be shipped by overnight courier to laboratory. Samples will leave the Site within 24 hours of sample collection, unless collected on a weekend. Samples collected on Saturday or Sunday will be shipped the following Monday.

In accordance with the AOC, the Region V USEPA RPM, Mr. Thomas Williams (telephone No. 312/886-6157), will be notified of all sampling activities at least 5 business days prior to commencing any sampling. The designated laboratory will be notified of sampling activities no less than 48 hours before sampling commences. This will ensure that the laboratory is ready to receive and process the samples within the time limits specified for the project.

FINAL SUPPORT SAMPLING PLAN

PART III

HEALTH AND SAFETY PLAN

for the

ENGINEERING EVALUATION AND COST ANALYSIS STUDY
OF THE FORMER CELOTEX SITE
2800 South Sacramento Avenue
Chicago, IL 60623

Prepared for:

ALLIEDSIGNAL, INC.
MORRISTOWN, NEW JERSEY
and
THE CELOTEX CORPORATION
TAMPA, FLORIDA

MARCH 1997

Prepared by:

PARSONS ENGINEERING SCIENCE, INC.
1000 JORIE BOULEVARD, SUITE 250
OAKBROOK, IL 60521

Parsons ES Project No. 730577

depending on the seriousness of the injury, decontamination may be disregarded:

- Life-saving care will be instituted immediately without considering decontamination procedure. The outside garments can be removed if they cause delays, interfere with treatment, or aggravate the problem. Respirators and backpack assemblies must always be removed after the person has been moved from the exclusion zone. Chemical-resistant clothing can be cut away. If the outer contaminated garments cannot be safely removed, the individual will be wrapped in plastic or blankets to help prevent contaminating medical personnel. A phone call notifying the hospital ER staff of the arrival of the individual wearing the contaminated garments will be made by the field team leader or the project health and safety representative. This procedure will help the ER staff prepare to receive the injured individual and will inform them of the presence of contaminated garments.

- For minor medical problems or injuries, the normal decontamination procedure will be performed.

9.2 CHEMICAL EXPOSURE

If a member of the field crew demonstrates symptoms of chemical exposure the procedures outlined below shall be followed:

- Another team member (*buddy*) shall remove the individual from the immediate area of contamination after the *buddy* has donned the appropriate PPE and has assessed that by removing the injured individual he would not be endangering himself. The *buddy* shall communicate to the field team leader (via two-way radio or hand signals) of the chemical exposure. The field team leader shall contact the appropriate emergency response agency.

- Precautions shall be taken to avoid exposure of other individuals to the chemical.

- If the chemical is on the individual's clothing, the chemical shall be neutralized or removed if it is safe to do so.

- If the chemical has contacted the skin, the skin shall be washed with copious amounts of water.

- In case of eye contact, eyes shall be washed with water or buffered eye wash solution for at least 15 minutes.